



NASA CR 120857

FINAL REPORT  
HIGH ENERGY DENSITY PRIMARY BATTERIES

by

R. J. Horning, Project Manager  
W. R. Beck  
E. M. Wroblewski

Honeywell Inc.  
Power Sources Center  
Route 309  
Montgomeryville, Pennsylvania 18936

prepared for

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

2 October 1972

Contract NAS-3-13221

NASA Lewis Research Center  
Cleveland, Ohio 44135  
William A. Robertson, Project Manager  
Direct Energy Conversion Division

Reproduced by  
NATIONAL TECHNICAL  
INFORMATION SERVICE  
US Department of Commerce  
Springfield, VA. 22151



(NASA-CR-120857) HIGH ENERGY DENSITY  
PRIMARY BATTERIES Final Report  
(Honeywell, Inc.) 277 P HC \$16.00

CSCL 10C

G3/03

Unclas  
54545

N73-16032

FINAL REPORT

HIGH ENERGY DENSITY PRIMARY BATTERIES

by

R. J. Horning, Project Manager  
W. R. Beck  
E. M. Wroblewski

Honeywell Inc.  
Power Sources Center  
Route 309  
Montgomeryville, Pennsylvania 18936

prepared for

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

2 October 1972

Contract NAS-3-13221

NASA Lewis Research Center  
Cleveland, Ohio 44135  
William A. Robertson, Project Manager  
Direct Energy Conversion Division

1. Report No. NASA - CR - 120857		2. Government Accession No.		3. Recipient's Catalog No.	
4. Title and Subtitle  HIGH ENERGY DENSITY PRIMARY BATTERIES				5. Report Date July 1971	
				6. Performing Organization Code	
7. Author(s) R. J. Horning, Project Manager; W. R. Beck; and E. M. Wroblewski				8. Performing Organization Report No.	
				10. Work Unit No.	
9. Performing Organization Name and Address Honeywell Inc. Power Sources Center Route 309 Montgomeryville, Penna. 18936				11. Contract or Grant No.  NAS 3-13221	
				13. Type of Report and Period Covered Contractor Report	
12. Sponsoring Agency Name and Address National Aeronautics & Space Administration Washington, D. C. 20546				14. Sponsoring Agency Code	
15. Supplementary Notes William A. Robertson, Project Manager, Direct Conversion Division, NASA-Lewis Research Center, Cleveland, Ohio 44135					
16. Abstract By the use of fractional factorials, paths of steepest ascent, and direct comparison experimentation, performance characteristics of the $\text{CuF}_2/\text{LiAsF}_6\text{-MF/Li}$ electrochemical system were optimized under conditions of temperature environment, discharge rate, active stand, and by the use of anhydrous cathodes. Two hundred ampere-hours/lb of $\text{CuF}_2$ and 600 watt-hours/lb of $\text{CuF}_2$ were achieved in anhydrous cells at $+40^\circ\text{C}$ and at a discharge rate of 10 ma/cm <sup>2</sup> . Electrical performance is primarily altered by temperature and discharge rate; however, other factors such as cathode water content, separator thickness, cathode carbon content, and the use of $\text{SO}_2$ gas also have significant effects.					
17. Key Words (Suggested by Author(s)) Primary Battery High Energy Density Battery Lithium Copper Fluoride			18. Distribution Statement  Unclassified/Unlimited		
19. Security Classif. (of this report) Unclassified		20. Security Classif. (of this page) Unclassified		21. No. of Pages 259	
				22. Price* 16.00	

\* For sale by the National Technical Information Service, Springfield, Virginia 22151

Details of illustrations in  
this document may be better  
studied on microfiche

## NOTICE

This report was prepared as an account of Government sponsored work. Neither the United States, nor the National Aeronautics and Space Administration (NASA), nor any person acting on behalf of NASA:

- A.) Makes any warranty or representation, expressed or implied, with respect to the accuracy, completeness, or usefulness of the information contained in this report, or that the use of any information, apparatus, method, or process disclosed in this report may not infringe privately owned rights; or
- B.) Assumes any liabilities with respect to the use of, or for damages resulting from the use of any information, apparatus, method or process disclosed in this report.

As used above, "person acting on behalf of NASA" includes any employee or contractor of NASA, or employee of such contractor, to the extent that such employee or contractor of NASA, or employee of such contractor prepares, disseminates, or provides access to, any information pursuant to his employment or contract with NASA, or his employment with such contractor.

## FOREWORD

The work described herein was performed at Power Sources Center, Honeywell Inc., under NASA Contract NAS 3-13221. The work was done under the management of the NASA Project Manager, Mr. William A. Robertson, Direct Energy Conversion Division, NASA, Lewis Research Center.

## High Energy Density Primary Batteries

by

R. J. Horning, Project Manager; W. R. Beck; and E. M. Wroblewski

### ABSTRACT

By the use of fractional factorials, paths of steepest ascent and direct comparison experimentation, performance characteristics of the  $\text{CuF}_2/\text{LiAsF}_6\text{-MF/Li}$  electrochemical system were optimized under conditions of temperature environment, discharge rate, active stand, and by the use of anhydrous cathodes. Two hundred ampere-hours/lb of  $\text{CuF}_2$  and 600 watt-hours/lb of  $\text{CuF}_2$  were achieved in anhydrous cells at  $+40^\circ\text{C}$  and at a discharge rate of  $10\text{ ma/cm}^2$ . Electrical performance is primarily altered by temperature and discharge rate; however, other factors such as cathode water content, cathode conductor content, separator thickness, and the use of  $\text{SO}_2$  gas also have significant effects.

## TABLE OF CONTENTS

	<u>Page</u>
List of Tables	viii
List of Figures	xii
Summary	1
1.0 Introduction	3
2.0 Compatibility Study	5
2.1 Preparation of Materials	5
2.1.1 Purification of Methyl Formate	
2.1.2 Recrystallization of $\text{KAsF}_6$	
2.1.3 Preparation of $\text{LiBF}_4$	
2.1.4 Electrolyte Preparation	
2.1.5 Control Data	
2.2 Separator Evaluation	7
2.2.1 Material Selection	
2.2.1.1 Testing Procedure	
2.2.2 Separator Resistance	
2.2.2.1 Resistivity Test Fixture	
2.2.2.2 Conductivity of Electrolyte	
2.3 Battery Case Compatibility Testing	
3.0 Cell Technology	11
3.1 Test Chamber Design	11
3.1.1 Standard Test Chamber	
3.1.2 Small Glass Test Chamber	

## TABLE OF CONTENTS (Continued)

		<u>Page</u>
3. 2	Experiment Program	12
3. 2. 1	Cell Fabrication	
3. 2. 2	Testing Program	
3. 2. 2. 1	Fractional Factorial - Experiments #1, 2, and 3	
	High Temperature-Low Discharge Rate Subregion	
3. 2. 2. 2	Steepest Ascent Vectors	
3. 2. 2. 2. 1	Experiments #4 and 6	
3. 2. 2. 2. 2	Experiment #7 - Copper vs. Silver Cathode Grids	
	Experiment #8 - Five- and Seven-Plate Cells	
	Experiment #9 - Performance vs. Electrode Surface Area	
	Experiment #13 - Performance vs. Discharge Rate	
3. 2. 2. 3	Moderate Temperature-High Discharge Rate vs. Subregion	
3. 2. 2. 3. 1	Experiment #11 - Adaptability of Exp. #6 Optimum to Subregion	
3. 2. 2. 3. 2	Experiment #12 - Fractional Factorial-Construction Variables	
3. 2. 2. 3. 3	Experiment #14 - Cathode Conductor, Binder and Separator Variation	
3. 2. 2. 3. 4	Experiment #15a and b	
	- Fractional Factorial-Construction Variables	
	Experiment #15c, d, and e	
	- Performance vs. Cathode Water and Separator Thickness	
3. 2. 2. 3. 5	Experiment #17 - Steepest Ascent Vector	
3. 2. 2. 3. 6	Experiment #24 - Steepest Ascent Vector	
3. 2. 2. 4	High Temperature-High Discharge Rate Investigations	
3. 2. 2. 4. 1	Experiment #16 - Separator, Carbon, Binder Variation	
3. 2. 2. 5	Performance Criteria	
3. 2. 2. 5. 1	Experiment #19a	
	- Establishing Levels of Primary Construction Variables	



## TABLE OF CONTENTS (Continued)

		<u>Page</u>
	Experiment #19b	
	- Effect of Temperature	
	Experiment #19c	
	- Effect of Electrolyte Molarity	
3.2.2.6	Anhydrous Cell Experiments	
3.2.2.6.1	Experiment #18b	
	- Initial Work	
	Experiment #20 - Effect of Cathode Conductor Content and Electrolyte Molarity	
	Experiment #21 - Effect of Separator and SO <sub>2</sub> Gas	
3.2.2.6.2	Experiment #22 - LiBF <sub>4</sub> Electrolyte Salt	
	Experiment #23 - Qualification Alternate Test Chamber	
	Experiment #26 - Performance vs. Discharge Rate and Temperature	
3.2.2.7	Active Stand Experiments	
3.2.2.7.1	Experiments #18a and 18c	
	- Effect of Separator Material and Thickness	
3.2.2.7.2	Experiment #25 Initial Work - Seven-Day Stand Use of SO <sub>2</sub>	
3.2.2.7.3	Experiment #27 - Anode Grid Influence on Active Stand	
	Experiments #28 and 29	
	- Effect of Electrolyte Volume on Active Stand	
	Experiment #31 Performance Comparison	
	- Alternate Material Sources	
3.2.2.7.4	Experiment #30 Active Stand vs. Storage Temp and SO <sub>2</sub> Quantity	
	Experiment #32 Effect of Electrolyte Age	
	Experiment #33 Active Stand vs. SO <sub>2</sub> Quantity	
3.2.2.8	Other Experiments (Exp. #5 and #10)	
3.3	Analysis of Cell Discharge Gases	45

## TABLE OF CONTENTS (Continued)

	<u>Page</u>
4.0 Summary of Results	47
Appendix "A" - Electrolyte Preparation Procedures PSC 8015-1	210
Appendix "B" - Karl Fischer Analysis Procedure PSC 8015-2	222
Appendix "C" - Compatibility Study Procedure PSC 8015-3	224
Appendix "D" - Resistance Measurement Procedure PSC 8015-4	228
Appendix "E" - Failure Analysis Procedure PSC 8015-5	231
Appendix "F" - Screen Cleaning Procedure PSC 8015-6	233
Appendix "G" - Preparation of Polystyrene/Xylene Solutions Procedure PSC 8015-7	235
Appendix "H" - Activation and Test Procedure PSC 8015-8	238
Appendix "I" - Cathode Fabrication Procedure PSC 8015-9	247
Appendix "J" - Anode Fabrication Procedure PSC 8015-10	249
Appendix "K" - Cell Construction Procedure PSC 8015-11	251
Appendix "L" - Chemical Analyses of Materials	253
Appendix "M" - List of Materials and Suppliers	262

## LIST OF TABLES

Table 1	Compatibility Data Summary of Separator Materials in 3.0-Molar LiAsF <sub>6</sub> /MF - Initial Series
Table 2	Compatibility Data Summary of Separator Materials in 3.0-Molar LiAsF <sub>6</sub> /MF - Second Series
Table 3A	Separator Material Resistance in 3.0-Molar LiAsF <sub>6</sub> /MF - Initial Series
Table 3B	Separator Resistance in 3.0-Molar LiAsF <sub>6</sub> /MF - Second Series
Table 3C	Compatibility Scan - Candidate Plastic Case Materials
Table 3D	Compatibility Scan - Candidate Plastic Case Materials
Table 4	Experiments #1 and #2 - Mean-Effect Data
Table 5	Experiment #1 - Discharge Data and Responses
Table 6	Experiment #2 - Discharge Data and Responses
Table 7	Experiment #3 Mean-Effect Levels - Yates Method
Table 8	Experiment #3 - Discharge Data and Responses
Table 9	Experiment #4 Cell Construction Characteristics
Table 10	Experiment #4 - Discharge Data and Responses
Table 11	Experiment #6 Cell Construction Characteristics
Table 12	Experiment #6 - Discharge Data and Responses
Table 13	Factor Levels for Experiments #7, #8, and #9
Table 14	Experiments #7, #8, and #9 - Discharge Data and Responses
Table 15	Experiment #13 - Discharge Data and Responses
Table 16	Experiment #11 Cell Construction Characteristics
Table 17	Experiment #11 - Discharge Data and Responses
Table 18	Experiment #12 Factors and Levels
Table 19	Experiment #12a Cell Construction Characteristics

## LIST OF TABLES

Table 20	Experiment #12a - Discharge Data and Responses
Table 21	Experiment #14 Cell Construction Characteristics
Table 22	Experiment #14a, b, and c - Discharge Data and Responses
Table 23	Experiment #14d - Discharge Data and Responses
Table 24	Experiment #14d - Mean-Effect Data
Table 25	Experiment #15a - Mean-Effect Levels - Yates Method
Table 26	Experiment #15a Cell Construction Characteristics
Table 27	Experiment #15a - Discharge Data and Responses
Table 28	Experiment #15b Cell Construction Characteristics
Table 29	Experiment #15b - Discharge Data and Responses
Table 30	Experiments #15c, d, and e Cell Construction Characteristics
Table 31	Experiment #15c, d, and e - Discharge Data and Responses
Table 32	Experiment #17 Cell Construction Characteristics
Table 33	Experiment #17 - Discharge Data and Responses
Table 34	Experiment #24 Cell Construction Characteristics
Table 35	Experiment #24 - Discharge Data and Responses
Table 36	Experiment #16 Cell Construction Characteristics
Table 37	Experiment #16 - Discharge Data and Responses
Table 38	Experiment #19 Cell Construction Characteristics
Table 39	Experiment #19a, b, and c - Discharge Data and Responses
Table 40	Anhydrous Cell Construction Variables
Table 41	Experiment #20 Cell Construction Characteristics

## LIST OF TABLES

Table 42	Experiment #20a, b, and c - Discharge Data and Responses
Table 43	Experiment #21 Cell Construction Characteristics
Table 44	Experiment #21 - Discharge Data and Responses
Table 45	Experiment #22 and #23 Cell Construction Characteristics
Table 46	Experiment #22 and #23 - Discharge Data and Responses
Table 47	Experiment #26 Cell Construction Characteristics
Table 48	Experiment #26 - Discharge Data and Responses
Table 49	Experiment #18a and c Cell Construction Characteristics
Table 50	Experiment #18a and c - Discharge Data and Responses
Table 51	Experiment #25 Cell Construction Characteristics
Table 52	Experiment #25 - Discharge Data and Responses
Table 53	Experiment #27 Cell Construction Characteristics
Table 54	Experiment #27 - Discharge Data and Responses
Table 55	Experiment #28 Cell Construction Characteristics
Table 56	Experiment #28 - Discharge Data and Responses
Table 57	Experiment #29 Cell Construction Characteristics
Table 58	Experiment #29 - Discharge Data and Responses
Table 59	Experiment #31 Cell Construction Characteristics
Table 60	Experiment #31 - Discharge Data and Responses
Table 61	Experiment #30 Cell Construction Characteristics
Table 62	Experiment #30 - Discharge Data and Responses

## LIST OF TABLES

Table 63	Experiment #33 Cell Construction Characteristics
Table 64	Experiment #33 - Discharge Data and Responses
Table 65	Experiment #32 Cell Construction Characteristics
Table 66	Experiment #32 - Discharge Data and Responses
Table 67	Experiment #5 Cell Construction Characteristics
Table 68	Experiment #5 - Discharge Data and Responses
Table 69	Experiment #5 - Mean-Effect Levels
Table 70	GC Analysis of Cell Discharge Gases

## LIST OF FIGURES

- Figure 1 Resistance of Multiple Layer Separators - Initial Series
- Figure 2 Resistance Test Fixture
- Figure 3 Conductivity Versus Molarity of  $\text{LiAsF}_6/\text{MF}$
- Figure 4 Cell Test Chamber
- Figure 5 Cell Test Fixture
- Figure 6 Glass Tube Cell Test Chamber
- Figure 7 Glass Tube Cell Test Fixture and Components
- Figure 8 Discharge Performance of Cells in Experiment #1
- Figure 9 Discharge Performance of Cells in Experiment #2
- Figure 10 Discharge Performance of Cells in Experiment #3
- Figure 11 Response Levels of Cells in Experiment #4
- Figure 12 Discharge Performance of Cells in Experiment #4
- Figure 13 Response Levels of Cells in Experiment #6
- Figure 14 Discharge Performance of Cells in Experiment #6
- Figure 15 Discharge Performance of Cells in Experiment #6
- Figure 16 Discharge Performance of Cells in Experiments #6 and #7
- Figure 17 Discharge Performance of Cells in Experiment #8
- Figure 18 Discharge Performance of Cells in Experiment #9
- Figure 19 Cathode Efficiency Versus Discharge Rate - Experiment #13
- Figure 20 Discharge Performance of Cells in Experiment #13
- Figure 21 Discharge Performance of Cells in Experiments #11 and #13
- Figure 22 Discharge Performance of Cells in Experiment #12a
- Figure 23 Discharge Performance of Cells in Experiment #12a

## LIST OF FIGURES

Figure 24	Discharge Performance of Cells in Experiment #14a
Figure 25	Discharge Performance of Cells in Experiment #14b
Figure 26	Voltage Versus Percent Cathode Efficiency - Experiment #14c
Figure 27	Discharge Performance of Cells in Experiment #14d
Figure 28	Discharge Performance of Cells in Experiment #14d
Figure 29	Discharge Performance of Cells in Experiment #14d
Figure 30	Discharge Performance of Cells in Experiment #14d
Figure 31	Discharge Performance of Cells in Experiment #15a
Figure 32	Discharge Performance of Cells in Experiment #15a
Figure 33	Discharge Performance of Cells in Experiment #15a
Figure 34	Discharge Performance of Cells in Experiment #15a
Figure 35	Discharge Performance of Cells in Experiment #15b
Figure 36	Discharge Performance of Cells in Experiment #15b
Figure 37	Discharge Performance of Cells in Experiment #15b
Figure 38	Discharge Performance of Cells in Experiment #15d
Figure 39	Discharge Performance of Cells in Experiment #15e
Figure 40	Response Level of Cells - Experiment #17
Figure 41	Discharge Performance of Cells in Experiment #17
Figure 42	Discharge Performance of Cells in Experiment #17
Figure 43	Discharge Performance of Cells in Experiment #17
Figure 44	Vector Performance - Experiment #24
Figure 45	Effect of Separator Layers - Experiment #24
Figure 46	Discharge Performance of Cells in Experiments #16a and #16b
Figure 47	Discharge Performance of Cells in Experiments #16c and #16d



## LIST OF FIGURES

Figure 48	Discharge Performance of Cells in Experiment #16e
Figure 49	Discharge Performance of Cells in Experiment #19a
Figure 50	Response Levels - Experiment #19a
Figure 51	Discharge Performance of Cells in Experiment #19b
Figure 52	Discharge Performance of Cells in Experiment #19c
Figure 53	Response Levels - Experiments #19b and #19c
Figure 54	Discharge Performance of Cells in Experiment #20b
Figure 55	Discharge Performance of Cells in Experiment #20c
Figure 56	Discharge Performance of Cells in Experiment #21
Figure 57	Discharge Performance of Cells in Experiment #22a
Figure 58	Discharge Performance of Cells in Experiment #22b
Figure 59	Discharge Performance of Cells in Experiment #23
Figure 60	Discharge Performance of Cells in Experiment #26a
Figure 61	Discharge Performance of Cells in Experiment #26b
Figure 62	Discharge Performance of Cells in Experiment #26c
Figure 63	Discharge Performance of Cells in Experiment #18a
Figure 64	Discharge Performance of Cells in Experiment #18a
Figure 65	Discharge Performance of Cells in Experiment #18c
Figure 66	Discharge Performance of Cells in Experiment #25a
Figure 67	Discharge Performance of Cells in Experiment #25b
Figure 68	Discharge Performance of Cells in Experiment #25c
Figure 69	Discharge Performance of Cells in Experiment #27
Figure 70	Discharge Performance of Cells in Experiment #27
Figure 71	Discharge Performance of Cells in Experiment #28
Figure 72	Discharge Performance of Cells in Experiment #29

## LIST OF FIGURES

Figure 73	Discharge Performance of Cells in Experiment #29
Figure 74	Discharge Performance of Cells in Experiment #29
Figure 75	Discharge Performance of Cells in Experiment #31
Figure 76	Discharge Performance of Cells in Experiment #31
Figure 77	Discharge Performance of Cells in Experiment #31
Figure 78	Discharge Performance of Cells in Experiment #30
Figure 79	Discharge Performance of Cells in Experiment #33
Figure 80	Discharge Performance of Cells in Experiment #32
Figure 81	Discharge Performance of Cells in Experiment #10

## SUMMARY

This report describes the work conducted on the  $\text{CuF}_2/\text{LiAsF}_6\text{-MF}/\text{Li}$  electrochemical system during the past 24 months.

Experiments were designed to map out the performance characteristics of the  $\text{CuF}_2/\text{LiAsF}_6\text{-MF}/\text{Li}$  system and its regions of optimum performance as a function of cell design and operational variables. Work included a compatibility and resistivity study of separator materials in three-molar  $\text{LiAsF}_6/\text{MF}$ . Additional experiments were conducted concerning the constituent materials for cell components.

As a result of the Task 1 compatibility studies, three materials were selected for use as separators in Task 2 experiments. Subsequent cell testing has shown glass fiber mat (Reeve Angel Grade 934H) to provide the best performance.

By the use of fractional factorials, paths of steepest ascent and direct comparison types of experiments, the performance characteristics of this system were optimized under conditions of low discharge ( $9\text{ ma/cm}^2$ ) and high temperature ( $+40^\circ\text{C}$ ); high discharge ( $38\text{ ma/cm}^2$ ) and moderate temperature ( $+10^\circ\text{C}$ ); high discharge ( $30\text{ ma/cm}^2$ ), high temperature ( $+40^\circ\text{C}$ ); anhydrous cathodes; and active stand. Yield values in terms of watt-hours/lb  $\text{CuF}_2$ , ampere-hours/lb  $\text{CuF}_2$ , and watt-hours/ $\text{cm}^3$  were used as a basis of evaluation.

Thirty-three experiments involving 702 tests were conducted. Results of these experiments have located areas of optimum performance. At a discharge rate of  $9\text{ ma/cm}^2$  and at a temperature of  $+40^\circ\text{C}$ , a yield was obtained of greater than 190 ampere-hours/lb of  $\text{CuF}_2$  (projected to better than 180 watt-hours/lb of battery).

Cells built to the same design point were quite sensitive to operational variables. At moderate temperatures (10-20°C), it was necessary to limit the current density to a maximum of 30 ma/cm<sup>2</sup> and to increase the amount of water in the cathode to at least 16 percent. Cells tested under these conditions yielded more than 190 ampere-hours/lb of CuF<sub>2</sub>.

At a discharge rate of 30 ma/cm<sup>2</sup> and at a temperature of +40°C, performance degraded to 140 ampere-hours/lb of CuF<sub>2</sub> despite the use of thicker, double layered separators to overcome the greater incidence of copper deposition in the separator.

Investigations into anhydrous cell technology produced the highest performance results of the program. Consistent yields of more than 200 ampere-hours/lb of CuF<sub>2</sub>, 600 watt-hours/lb of CuF<sub>2</sub> at 10 ma/cm<sup>2</sup>, and +40°C were obtained. Anhydrous cells are extremely sensitive to discharge temperature, performing best between +35°C and +40°C. However, limited experimentation with SO<sub>2</sub> gas in the cell improved +30°C performance dramatically.

In 7-day and 14-day active stand investigations, distinct performance improvements resulted from the introduction of SO<sub>2</sub> gas in the cell. Cell efficiencies at 7-day active stand were raised from less than 16 percent without SO<sub>2</sub> gas, to more than 69 percent with SO<sub>2</sub>. In active stand testing, silver leads and grids for the anode corroded rapidly and, therefore, silver was replaced by the more suitable copper.

A cell was designed and successfully operated to a prorated specific electrical requirement as a test of the validity of the techniques and of the results obtained in this program. The requirement called for discharge at 80 amperes with a 150 ampere-hour capacity. The cell was designed on the basis of operating at 20 ma/cm<sup>2</sup>.

## 1.0 Introduction

This final report describes the experimental work performed during the period of 20 June 1969 to 30 June 1971 under Contract NAS 3-13221 for NASA-Lewis Research Center toward the further development of high energy density primary batteries. This program is a continuation of work performed under Contract NAS 3-10613 (Final Report NASA CR-72535) and earlier contracts for the development of a high energy density primary battery. The purpose of this particular phase of the general program was to map out the performance characteristics of the  $\text{CuF}_2/\text{LiAsF}_6\text{-MF/Li}$  battery system, which has been identified as suitable for high rate, primary reserve applications. It was further intended to determine regions of optimum performance as a function of cell design and operational variables.

The scope of the program was originally defined in terms of three tasks. Task 1 was a compatibility and resistivity study of separator materials, the result of which provided candidate materials for use in subsequent cell testing. Task 2 was the major portion of the program and was a general investigation of the system's performance relative to operational and constructional variables. Task 3 was to use the design information, procedures, and fabrication techniques generated in Task 2 to construct 20 demonstration cells having optimum design features and the largest feasible capacity. However, the scope of the program was changed during its second year to eliminate the construction of the 20 demonstration cells and to replace them with an active stand life characteristic, cell performance study.

The initial experimentation in Task 2 relied heavily on statistical disciplines concerning factorial and fractional factorial designs. The experimental plan assumed that the dependent variables of ampere-hours/lb of  $\text{CuF}_2$ , watt-hours/lb, and watt-hours/cm<sup>3</sup> are a continuous function of the independent variables chosen, both operational and constructional. It was further assumed that the functions could be

approximated by first and second-order polynomials that have identifiable maximums in specific areas of variation of the factors involved. The first experiments were run in a selected subregion and the resultant data fitted to a general regression equation by the method of multiple linear regression. This particular phase of the work was conducted at the NASA-Lewis Research Center through the use of the Rapier computer program. When it had been decided the resultant regression equation had provided sufficient fit in the chosen subregion, the equation coefficients were used to determine vectors of steepest response. These vectors were studied until it could be determined that a maximum had been achieved in the particular subregion being investigated. Experimental points around this maximum design were then taken in an effort to map out the response function in more detail. Throughout the procedure, variables that indicated nonsignificant coefficients after a sufficient amount of study were disregarded in further experimentation.

The foregoing experimental procedure was used in determining the maximum design point for the high temperature, low discharge rate performance region and the moderate temperature, high discharge rate performance region. In the region of high temperature, high discharge rates and in the anhydrous and active stand studies this statistical strategy was replaced by the more conventional experimental technique of direct comparison testing.

The areas of involvement related to this program are significant in their further development of general lithium-oriented battery technology. The data generated can provide the background necessary to enable a practical application of these high energy output cells with active stand capability, at a considerable saving in weight and volume over many systems currently available.

## 2.0 Compatibility Study

This section of the report discusses the work performed during Task 1, separator evaluation. It also includes the preparation of electrolyte and its constituent materials, which are also used in the Task 2 phase of the program.

### 2.1 Preparation of Materials

Preparation of three-molar lithium hexafluoroarsenate in methyl formate<sup>1</sup> (LiAsF<sub>6</sub>/MF) electrolyte was necessary to conduct Task 1. Materials used in this electrolyte, as received from vendors, were initially analyzed and then further processed to provide control over the quality of the materials used. Where feasible, materials were purchased as a single lot to ensure uniformity. Special handling and storage procedures were developed and employed to prevent contamination during the program life.

Each of the "as received" materials were analyzed for impurities and water content. Spark source and emission spectroscopy were used on impurity analysis of lithium tetrafluoroborate (LiBF<sub>4</sub>), potassium hexafluoroarsenate (KAsF<sub>6</sub>), and copper fluoride (CuF<sub>2</sub>). X-ray defraction was used to determine water in CuF<sub>2</sub> samples periodically. Karl Fischer techniques were used to determine water in LiBF<sub>4</sub> and KAsF<sub>6</sub>. Gas chromatography (GC) was employed in the analysis and control of the methyl formate (MF).

Anhydrous CuF<sub>2</sub>, double treated with hydrogen fluoride (HF), was ordered from Ozark-Mahoning. Thirty-five pounds in one-pound containers were received as one lot, KW-6-133. This material was stored in air-tight containers with a standard desiccant. Other materials received from Ozark were ten pounds of copper fluoride dihydrate (CuF<sub>2</sub>·2H<sub>2</sub>O), Lot No. WH-1-86D, and 50 pounds of

<sup>1</sup> Electrolyte of other molarity was required in Task 2 responsibilities; however, the manufacturing procedure was essentially the same.

KAsF<sub>6</sub>, Lot No. BD-1-24. The LiBF<sub>4</sub> (ten kilograms), Lot No. 808-1, was received from Foote Mineral, as was all the lithium metal used in the program. The methyl formate (MF) us spectroquality material obtained from Matheson, Coleman and Bell.

#### 2.1.1 Purification of Methyl Formate

Purification of the MF consisted initially of filtering the raw stock through a Linde 4A molecular sieve and agitating the resulting filtrate with powdered lithium. This solution was then distilled at a flow rate of 6.5 ml/min, and the first 400 cc of an 1800 cc batch was discarded and the remaining collected in argon-filled bottles which were vented to the air through a drying tube packed with Linde 4A molecular sieve. The distilled solution was analyzed by GC for water, methanol, and any other impurity present in greater than 10 parts/million concentration.

#### 2.1.2 Recrystallization of KAsF<sub>6</sub>

Purification of this component salt from its raw stock condition was accomplished by recrystallization from distilled water. Approximately 720 grams of KAsF<sub>6</sub> were dissolved in 2000 ml of distilled water and then heated to +60°C. The solution was filtered, and the collected filtrate evaporated to two-thirds of its original volume by heating to +100°C. The solution was then allowed to recrystallize for a minimum of 12 hours, after which the crystals were separated from the solution via suction filtration. The salt was then placed under a soft vacuum, 26" to 28" of mercury, for one hour and a vacuum of 29" to 30" cf mercury, over concentrated H<sub>2</sub>SO<sub>4</sub> for 48 hours. The salt was then dried for a minimum of 48 hours at +110°C in a vacuum oven. The product was analyzed by Karl Fischer for water content.



### 2.1.3 Preparation of $\text{LiBF}_4$

The "as received" material was dried in a vacuum oven for a minimum of 48 hours at  $+110^\circ\text{C}$ . The material was then checked for water content by the Karl Fischer technique.

### 2.1.4 Electrolyte Preparation

The electrolyte required for both Task 1 and 2 was prepared by dissolving the  $\text{KAsF}_6$  in MF and then adding  $\text{LiBF}_4$  to the resulting solution. The resultant insoluble reaction product ( $\text{KBF}_4$ ) was then checked stoichiometrically against the theoretical amount which should have been precipitated, and based on this comparison a determination of reaction completion was made. The solution was then adjusted either by evaporation or dilution to the proper molarity. A detailed description of this procedure can be found in Appendix "A," page 208.

### 2.1.5 Control Data

Each of the materials used in the manufacture of the electrolyte was analyzed for impurities and water content. Forty-five batches of electrolyte were used. Water content, determined by the Karl Fischer method, ranged from 90 ppm to 260 ppm. The nominal range for 80 percent of the batches was 120 to 180 ppm.

Values obtained for  $\text{H}_2\text{O}$  and  $\text{MeOH}$  content in methyl formate, as determined by GC analysis, ranged from less than 10 ppm to around 90 ppm. Batches with greater than 100 ppm concentration of either of these impurities were rejected.

## 2.2 Separator Evaluation

### 2.2.1 Material Selection

Materials selected for the compatibility scan in three-molar  $\text{LiAsF}_6/\text{MF}$  included materials previously used in this cell system, those recommended by NASA, and other promising materials selected from a preliminary scan of candidate types in two-molar  $\text{LiAsF}_6/\text{MF}$  that resulted from a review of those available in the field. Six candidate materials were selected on the basis of an initial review and subjected to the first complete series of compatibility tests in three-molar  $\text{LiAsF}_6$ . The three materials selected from the initial series of tests were Glass Fiber Mat (Reeve Angel Grade 934H), polypropylene (Pellon FT2140), and Dacron (Kendall Weblox EV100). The Kendall material was later replaced by DuPont "Tetryls" Dacron material. This replacement was necessary because the Weblox material could not conform to the separator thickness levels chosen for experimentation. The substituted material exhibited similar compatibility and resistivity results and met the thickness specification. Unfortunately, when an attempt to obtain additional quantities for use in cell testing were made, it was discovered the product line had recently been discontinued and was no longer available. This made it temporarily necessary to proceed with cell testing with only two candidate separator materials. At a later time during the cell technology phase, a second series of compatibility and resistivity tests were conducted on materials of the rayon, dacron, polypropylene, and combinations thereof to select a third separator material. As a result of this study Rayon Polypropylene Carded E4208 and Dacron E1486 were selected. A final comparison of performance characteristics was accomplished in the cells tested in Experiment #5.

#### 2.2.1.1 Testing Procedure

The materials selected for the compatibility scan were cut into 1" x 2" strips. Each strip was carefully weighed and measured and its general appearance noted. The strips were then placed into a compatibility fixture and charged with 30 cc

of electrolyte. One fixture was charged with electrolyte only, to serve as control, and placed under test along with the separator samples. During the 100 hours at +50°C test any changes in physical appearance of the samples and electrolyte were noted at 24-hour intervals. At the conclusion of the test cycle, the containers were removed from the environment and allowed to cool. The samples were rinsed in pure MF until clean (determined by a conductivity measurement of the MF rinse) and dried under vacuum for 15 hours. The samples were then checked for changes in weight, size, and physical characteristics from the original values taken. A summary of compatibility data is shown on Tables 1 and 2.

### 2.2.2 Separator Resistance

Each of the separator materials in single and multiple layers was tested to determine its electrical resistance in three-molar  $\text{LiAsF}_6/\text{MF}$  at +30°C. It was found, as would be expected, that in time the separators became saturated with electrolyte and their resistances decreased significantly. This decrease in resistance was more pronounced in multiple layered separators. Readings were taken at initial immersion, after five minutes, and after 100 hours. Resistance readings after 100 hours did not change significantly from the five-minute values. See Figure 1 and Tables 3a and 3b for the first series results.

#### 2.2.2.1 Resistance Test Fixture

The fixture used for the resistance measurements consisted of two glass elbows, platinum electrodes, and a frame as shown in Figure 2. To determine the resistance of the materials, three-molar electrolyte was added to the unit and the resistance measured; then the separator material, retained in a rigid frame, was added and another measurement taken. The difference in readings indicated the resistance of the material being evaluated.

#### 2.2.2.2 Conductivity of Electrolyte

Additional data referencing the molarity of the electrolyte is given in Figure 3.

#### 2.3 Battery Case Compatibility Testing

Prior to the elimination of Task 3, which called for the construction of 20 demonstration models, tests were carried out to determine the compatibility of several candidate rubber and plastic case materials with the electrolyte. Of the six rubber materials tested, ethylene propylene was most compatible and of the 16 plastic materials tested high density polyethylene, polypropylene, and glass filled polyester rated best. The results of the compatibility scans are shown in Table 3c.

### 3.0 Cell Technology

This section of the report discusses the work conducted as part of Task 2.

#### 3.1 Test Chamber Design

##### 3.1.1 Standard Test Chamber

As part of the Task 2 responsibility it was necessary to design and manufacture 20 test chambers for use in experimentation. The required test chamber was designed for maximum flexibility in the test program. Through the use of reducing frames it would allow for the three sizes of cells to be tested using the same basic outer structure. Included in the design were pressure and temperature monitoring ports as well as a pressure relief valve for safety purposes. The chamber and its component parts are illustrated in Figures 4 and 5.

The chamber is constructed of high density polyethylene material backed by aluminum plates, which provide additional rigidity and structural strength. The chamber compartment is sealed by an "O" ring to prevent the escape of pressure and electrolyte. The chamber is designed to allow testing up to 100 psig.

Temperature is monitored by a copper/constantan thermocouple positioned in a blind hole at one of the wall surfaces of the chamber. Pressure is recorded through the use of a suitable transducer connected to one of the monitoring ports in the top of the unit. Voltage monitoring is achieved through the two terminals positioned on either side of the main cell compartment.

##### 3.1.2 Glass Tube Cell Chambers

An amendment to the contract that replaced Task 3 with work directed toward the study and identification of system capabilities in the active stand area necessitated the use of an additional test vehicle. The test chamber chosen for the majority of the active stand investigation was a standard aerosol compatibility vessel supplied by Fisher and Porter. The chamber and its component parts are illustrated in Figures 6 and 7.

The tube is fitted with standard stainless steel hardware and a needle valve adapter. The standard "O" ring seal supplied with the needle valve adapter was replaced with one of ethylene polypropylene for better compatibility. The gauge is used to monitor pressure during the active stand period.

Three No. 26 teflon coated wires are inserted into the tubes through Swagelok fittings and connected to the cell plates (two anodes and one cathode) via a solder connection. A plastic insulator is fitted over this connection to prevent accidental shorting during the testing. These wires are used to monitor voltage during test.

### 3.2 Experimental Program

During the experimental program, 33 experiments consisting of approximately 700 cells have been designed, tested, and analyzed. The first of these experiments were used to determine regions of optimum cell performance as a function of cell design and operational variables using statistical strategy. In the latter part of the program and at varied points early in the program, experiments were conducted for specific points of information.

The initial 15 experimental factors selected for investigation were: operating temperature, discharge rate, separator material, separator thickness, electrolyte concentration, electrolyte volume, electrode area, collector screen geometry, number of electrodes/cell, anode thickness, cathode thickness, cathode water content, cathode conductor material, cathode conductor content, and cathode binder content.

Initial experiments conducted were fractional factorials, which estimate the first-order effects of the 15 factors, provide the basis for later path of steepest ascent experiments, and then eventually lead to a maximum design point in a selected subregion. Other experiments provide direct comparison to evaluate new materials or cell construction techniques.

Data generated by the fractional factorial experiments were analyzed by the Yates method of calculation of effects and mean squares described on page 263 of Design and Analysis of Industrial Experiments by Davies, and by means of NASA's Rapier computer program. From the data generated, areas of optimum operation were defined and sensitivity to discharge rate in terms of performance were investigated. Later emphasis was placed on developing maximum design points somewhat displaced from the "floating" optimum in terms of temperature and discharge rate. The optimum point determined was located in the low discharge (nine ma/cm<sup>2</sup>) and high temperature (+40°C) region.

### 3.2.1 Cell Fabrication

Fabrication of individual components used in each experiment was performed in a dry room with the relative humidity controlled and maintained at less than three percent. Additional precautions taken to insure consistent ambient conditions and limited contamination included working within an argon-filled glove box, purged on a regular basis, which maintained the H<sub>2</sub>O level at less than 0.5 mg of H<sub>2</sub>O/liter of gas. This glove box was used in the preparation of the cathodes.

Cathode and anode molds were initially fabricated to cover the three cell sizes and the various thicknesses needed.

The negative electrodes were prepared by placing the proper weight of lithium sheet and expanded metal into the mold and pressing with sufficient pressure

[ $6.90 \times 10^6$  newton/meter<sup>2</sup> (1000 psig)] to insure an adequate meshing of the material and screen. The finished components were stored in a vacuum until fabricated into the cells.

The positive plates were constructed by the pasting of a  $\text{CuF}_2$ ,  $\text{CuF}_2 \cdot 2\text{H}_2\text{O}$ , carbon, styrene, xylene mixture to silver grids. The amount of binder content, conductor material, and water was dependent on the experimental design. The pasting process was done within an argon-filled glove box. The amount of water vapor in the box was minimized by a recirculating drying train, and the atmosphere was analyzed periodically by GC for water vapor.

### 3.2.2 Testing program

Once the necessary controls and procedures had been implemented and verified through trial cell tests, Task 2 was initiated with the testing of Experiment #1. Each succeeding experiment was based on the result of preceding tests, with possible exceptions where new materials or construction techniques were being tested. All cells, except those tested under constant current conditions in Experiment #14c, were discharged through a fixed resistor. The current densities were calculated using a nominal 3.0-volt cell voltage.

#### 3.2.2.1 Fractional Factorial - Experiments #1, #2, and #3

Experiment #1 was a fractional factorial experiment having 15 independent variables. There were 16 different cells, four of which were replicated for the purpose of establishing experimental error. The factor levels for this experiment are listed below.



Factor Levels for Experiments #1 and #2		Low Level	High Level
A	Discharge Temperature	+10° C	+30° C
B	Discharge Rate	20 ma/cm <sup>2</sup>	30 ma/cm <sup>2</sup>
C	Separator Material	Reeve Angel Glass	Pellon FT2140
D	Separator Thickness	.015"	.040"
E	Electrolyte Concentration	2.0 Molar	3.0 Molar
F	Electrolyte Volume	100% Full	110% Full
G	Electrode Size	2" x 3"	2-3/4" x 4"
H	Screen Geometry	Standard Exmet 5Ag 14-1/0	Standard Distex 5Ag 14-1/0
J	No. of Electrodes	3	5
K	Anode Capacity	110% of Cathode	125% of Cathode
L	Cathode Thickness	.040" Nominal	.080" Nominal
M	Cathode H <sub>2</sub> O Content	4%	8%
N	Cathode Conductor Material	Conductex	Asbury
O	Cathode Conductor Content	5%	10%
P	Cathode Binder Content	1%	2%

The fundamental identity for developing the defining contrast is as follows:

$$I = ABE = ACF = ADG = BCH = BDJ = CDK = ABCL = ABDM = ACDN = BCDO = ABCDP$$

The complete identity is achieved by multiplying each term by every other term. In Experiment #1 the principal block of factor combinations was used. In Experiment #2 another block of factor combinations was employed. References on the statistical methodology can be found in Design and Analysis of Industrial Experiments, Owen L. Davies, ed., Hafner Publishing Company, New York, Second Edition, 1960.

The data which resulted from this experiment were analyzed by the NASA Rapier program and by the Yates analysis method. A ranking of responses according to absolute value is given in Table 4.

The analysis of Experiments #1 and #2 involved comparison of the mean-effect data resulting from responses calculated on the basis of ampere-hours/lb of  $\text{CuF}_2$  ( $y_2$ ), watt-hours/lb ( $y_4$ ), and watt-hours/ $\text{cm}^3$  ( $y_6$ ). These mean-effect data represent coefficients of a regression equation that approximate the area of the response function investigated. The analysis of the data determined for each factor the level which had the greatest positive effect on battery performance. This then implied, for quantitative factors, a direction in which to change the value of that factor. The following lists the indications provided by the analyses:

Factor		Indication
A	Discharge Temperature	Increase above $+20^\circ\text{C}$
B	Discharge Rate	Decrease below $20 \text{ ma/cm}^2$
C	Separator Material	Glass preferred over Pellon
D	Separator Thickness	Decrease below $.0275''$
E	Electrolyte Concentration	Decrease below 2.5 molar
F	Electrolyte Volume	Insignificant for range investigated
G	Electrode Size	$2\text{-}3/4'' \times 4''$ preferred over $2'' \times 3''$
H	Screen Geometry	Distex preferred over Exmet
J	No. of Electrodes	Increase to 5 or more
K	Anode Capacity	Increase to greater than 110%
L	Cathode Thickness	Increase to above $.060''$
M	Cathode $\text{H}_2\text{O}$ Content	Increase to above 6%
N	Cathode Conductor Material	Asbury Ceylon preferred over Conductex SC
O	Cathode Conductor Content	Increase above 7.5%
P	Cathode Binder Content	Increase above 1.5%

It can be noted that not all the above indications carried the same weight. Some of the factors, such as A, B, C, D, and E, were very strong, whereas some of the others were relatively weak. The relative strength of each indication can be best judged by comparison of the mean-effect data listed on Table 4.

The discharge data for Experiments #1 and #2 are listed on Tables 5 and 6, and representative discharge curves are given in Figures 8 and 9. It is difficult to make direct comparison between specific cells within an experiment on the basis of one factor or another, since each cell is different according to statistical design. The comparison can only be effected when all the factors are considered together, as is done in the Yates or computer analysis. For this reason only representative curves have been provided.

In the design of Experiment #3, the indications provided for in previous experimentation and their relative strength were used to establish the new factor levels. This information was also used to establish those which were not highly significant, which in turn could be held constant in the next experiment. The factor levels for Experiment #3 were as follows:

Factor		Levels
A	Discharge Temperature	+20°C and +40°C
B	Discharge Rate	10 ma/cm <sup>2</sup> and 20 ma/cm <sup>2</sup>
C	Separator Material	Glass and Pellon
D	Separator Thickness	0.015" and 0.030"
E	Electrolyte Concentration	1.7 molar and 2.7 molar
F	Electrolyte Volume	10% excess (for all cells)
G	Electrode Size	2-3/4" x 4" (for all cells)
H	Screen Geometry	Exmet and Distex
J	No. of Electrodes	3 (for all cells)
K	Anode Capacity	125% (for all cells)
L	Cathode Thickness	0.040" and 0.100"
M	Cathode H <sub>2</sub> O Content	2% and 6%
N	Cathode Conductor Material	Conductex and Asbury
O	Cathode Conductor Content	7% and 12%
P	Cathode Binder Content	1% and 2%

The results of Experiment #3 show that some improvement had been achieved in terms of the yield values. Ampere-hours/lb of CuF<sub>2</sub> values approached the 200 level, which represented 80% to 85% of cathode efficiency. This provided the general indication that the factor changes made for this experiment were creating a positive effect. The mean-effect data shown on Table 7 indicates that the four most significant factors affecting cathode efficiency were temperature, discharge rate, separator material, and percent cathode H<sub>2</sub>O. For the other two yield values the relative ranking changed, indicating that other factors took on greater significance. This is best described in Table 7, which lists all the mean-effect data for this experiment. Discharge data are shown on Table 8 and representative curves on Figure 10.

### 3.2.2.2 Steepest Ascent Vectors

From the background provided in the first three experiments, which located the general area of the optimum subregion, it was now possible to investigate several vectors of steepest ascent. This was done in Experiments #4 and #6.

#### 3.2.2.2.1 Experiments #4 and #6

In Experiment #4 temperature, discharge rate, and electrolyte concentration were the variable factors. The remaining factors were held constant. The range of variations and the levels of all the factors are described in Table 9.

The resulting data generated in this experiment reflect no true maximum; however, there were some areas of improvement in yield values. Figure 11 plots the response values against test combinations. Discharge data and selected curves are given on Table 10 and Figure 12.

A second steepest ascent vector was investigated in Experiment #6. However, in this experiment six factors were varied--temperature, discharge rate, electrolyte concentration, separator thickness, cathode water, and cathode conductor content. The ranges of these and the levels of the remaining factors held constant are given in Table 11.

There were five test combinations of factors in Experiment #6, and three cells at each point were run. Increases in yield in terms of watt-hours/lb, watt-hours/cm<sup>3</sup>, and ampere-hours/lb of CuF<sub>2</sub> were obtained through each successive combination up to the fourth set. The No. 5 combination produced a reduction in yield from the No. 4 position. A chart of the yields versus test factors is given in Figure 13 and the discharge data on Table 12. Discharge curves for these tests are shown in Figures 14 to 16.

The results obtained in terms of yield values (attaining 203 ampere-hours/lb of  $\text{CuF}_2$ ) were the largest obtained for three-plate cells to this time, and they represented either a maximum or a plateau in the yield surface.

#### 3.2.2.2.2 Experiments #7, #8, #9, and #13

It was decided at this time to consider the combination at the No. 4 point in Experiment #6 as a maximum design point for the higher temperature, low discharge rate subregion. In Experiments #7, #8, #9, and #13 additional data were generated that would reflect some of the sensitivities of this maximum design point to several specific factors. This was done to provide a more detailed map of the particular subregion of the response surface under investigation. The cell character grid for these four experiments is shown on Table 13, the discharge data on Table 14, and the discharge curves on Figures 16 through 18.

Experiment #7 was performed to determine if copper could be substituted for the silver used as a cathode grid, since copper has distinct advantages in cost and weight. Resultant yield values were slightly lower for the cells using the copper; however, the difference from the cells using the silver grid was not significant enough to disqualify its use if material became of primary importance.

Experiment #8 was a two-part experiment to determine sensitivity of the maximum design point to the number of plates in the cell. Yield values (in terms of watt-hours/lb and watt-hours/cm<sup>3</sup>) should increase as the number of plates are increased, since the ratio of capacity-to-total weight and total weight and total cell volume are not constant. Two 5-plate cells and two 7-plate cells were run in this experiment. Increases in yield were obtained for each increase in the number of plates. The maximum watt-hour/lb value obtained to the 2.0-volt level was 180 for seven-plate cells. The data indicated that an approximate increase in watt-hours/lb and watt-hours/cm<sup>3</sup> of 15 percent is obtained for each additional cathode in the cell up to seven plates.

There is no reason not to expect this advantage to continue for cells containing numbers of plates greater than we have tested, for the weight and volume advantage is continued with each successive cathode. However, there should be a point where other influences (primarily mechanical) such as activation, heat, etc., will begin to reduce and eventually eliminate this advantage. This being the case, an optimum in terms of the number of plates should exist. At this point it can be stated only that this optimum is at a number greater than seven.

Experiment #9 was conducted to determine if yields obtained at the fourth combination level in Experiment #6 would be valid for a cell with smaller effective areas. Three 6-square-inch cells (2" x 3") instead of the 11-square-inch cells (2-3/4" x 4") were discharged. Yield values were at the same level for ampere-hours/lb of  $\text{CuF}_2$  and watt-hours/ $\text{cm}^3$ ; however, the watt-hour/lb values were reduced. The watt-hour/lb yield value is largely dependent on the amount of electrolyte used, which makes up the major portion of the cell weight. There is a certain amount of void-volume within the chamber that increases the amount of electrolyte a cell can contain. As the cell size decreases, the void area becomes a larger percentage of the total volume, thus having the effect of decreasing the watt-hour/lb values for smaller cells. Correction factors were employed based on the amount of void; however, values still remained somewhat lower than for the larger cells, indicating there may be a loss in yield in terms of watt-hours/lb as cell size is decreased.

In Experiment #13 the sensitivity of the maximum design point to the level of discharge rate was investigated. The cells used had the same construction as Experiment #6, position No. 4 and were discharged at +40°C. Six cells were discharged, two each at 10, 20, and 30 ma/ $\text{cm}^2$ . Figure 19 plots the cathode efficiency against discharge rate and indicates the losses incurred as the latter is increased. Discharge data for this experiment is listed on Table 15 and discharge curves on Figures 20 and 21.

### 3.2.2.3 Moderate Temperature and High Discharge Rate Subregion

The experiments described thus far are the extent of the investigation into the high temperature, low discharge subregion. Also of interest is the cell's performance in the low and moderate temperature range and higher discharge rates. Investigation into this area was undertaken with Experiment #11.

#### 3.2.2.3.1 Experiment #11

Experiment #11 has investigated a set of variables similar to those treated in Experiment #6, with the exception that operational variables (temperature and discharge rate) have been held constant at  $+15^{\circ}\text{C}$  and  $30\text{ ma/cm}^2$ , respectively. The factors varied were separator thickness, electrolyte concentration, cathode thickness, and percent cathode  $\text{H}_2\text{O}$ . Each combination of variables has been tested in triplicate. The experimental format is given in Table 16 and the discharge data on Table 17. A representative curve is shown on Figure 21.

The performance of the cells in this experiment have been generally poorer than that obtained in previous experiments, with the exception of some earlier low temperature, high discharge rate cells run in Experiments #1 and #2. The data have indicated increases in yield for each successive group of test factors, maximizing at the No. 4 group for ampere-hours/lb of  $\text{CuF}_2$  and the No. 3 group for watt-hours/lb and watt-hours/ $\text{cm}^3$ . The range of the yields for duplicate cells at each point was quite wide and reflects basic instability in the range of values that were treated.

Experiment #11 has demonstrated that the maximum design point obtained in Experiment #6 is not applicable in the low temperature, high discharge subregion and is quite sensitive to the changes in operational variables. Since construction variables were not significantly changed from the Experiment #6,

step No. 4 position, the degradation in performance can be primarily attributed to the changes in temperature and discharge rate. This experiment indicates the possibility of optimum subregions in the response surface quite apart from one another depending on the position along the temperature axis being investigated.

### 3.2.2.3.2 Experiment #12

The format of Experiment #12, which continued investigation into the low temperature, high discharge subregion, returned to the initial stages of the statistical procedure where the selected subregion was investigated in terms of construction variables by fractional replicate of  $2^n$  factorial experiments.

Originally the plan was to run Experiment #12 in two parts. Part "A" was a one-half fractional factorial experiment of 16 cells that would vary five factors at two levels, represented in the L and H columns on Table 18. In addition, this experiment had six cells having a center point combination of factors, which was to aid in the estimation of experimental error and confidence levels in the results obtained. All the 16 L and H cells were tested in duplicate. Part "B" of the experiment was to have been a test of 10 star points (factors represented by  $[S^-$  and  $S^+]$  in the following table) and an additional four cells at the center point.

TABLE 18

#### Experiment #12 Factors and Levels

	( $S^-$ )	L	<u>Levels</u> C. Pt.	H	( $S^+$ )
Separator Thickness	.015	.020	.025	.030	.035
Electrolyte Molarity	1.8 M	2.1 M	2.4 M	2.7 M	3.0 M
Cathode Thickness	.025	.035	.045	.055	.065
Cathode H <sub>2</sub> O Content	2%	4%	6%	8%	10%
Cathode Conductor Content	5%	7%	9%	11%	13%



The yields of Experiment #12a were unexpectedly poor so that Part "B" of Experiment #12 was deferred and ultimately replaced by Experiment #15 after the interactions of Experiment #14 shifted the values from the levels shown in Table 18 to those in Table 24. Cell construction details and discharge data for Experiment #12a are shown in Tables 19 and 20, respectively. Several discharge curves are shown in Figures 22 and 23.

The voltage levels of Experiment #12a were depressed below the 2.0-volt minimum in many cases but, several units discharged quite efficiently above 1.0 volts; therefore, the discharge data were used for calculations to both the 2.0-volt and 1.0-volt levels. One of the indications from the interactions is the apparent dependency of the three factors treated upon the separator thickness. The level of each of these factors becomes more important as the thickness of the separator material increases. Therefore, by maintaining the separator thickness at .020" or possibly lower, at these temperatures, it was not necessary to displace the level of the other factors to any large degree. This is borne out in Experiment #14.

#### 3.2.2.3.3 Experiment #14

In Experiment #12a, Cells F-222 and F-223 had factors at the levels preferred in the Yates analysis of the experiment and were, on the average, the best performing cells of the experiment. Therefore, in the design of Experiment #14, whose primary objective was to generally improve the moderate temperature, high discharge cell performance, the same combination of factors was used except for the factors under investigation. It was found in Experiments #14a and #14b that the use of Conductex SC in the cathode, lowering of the percentage of styrene binder in the cathode, and reducing the separator thickness contributed to a slightly increased efficiency at the 1.0-volt level (see Figure 24). Previously Asbury material was chosen in preference to Conductex, primarily on the basis of its handleability and the structural strength it imparted to the cathodes. It appeared

now, however, that cell performance at this moderate temperature subregion, particularly the low voltage at the beginning of the test, benefited from the highly cracked condition prevalent in the cathodes made from Conductex. This is shown in Figure 25. The details of cell construction for Experiment #14 and resulting data from Experiment #14a, b, and c are shown in Tables 21 and 22.

In addition to the normal constant resistance method of discharging the cells, in Experiment #14c several cells were discharged at constant current. This was done to determine if any difference in cell performance was imparted as a result of the mode of testing. The discharge data indicated that a higher level of power could be sustained in the constant "I" mode; however, these cells were less efficient than those run at constant "R". Testing of constant "I" cells also revealed a sensitivity of the output voltage to small changes in current density. This was evidenced primarily in Cell F-256 where several changes were made in current during the run. Initially this cell could not support 38 ma/cm<sup>2</sup>; however, it discharged rather well at 30 ma/cm<sup>2</sup>. It continued to do so until the current was increased to 33 ma/cm<sup>2</sup>, at which point the voltage level began to decay rapidly. This is illustrated in Figure 26. Experiment #14c was important because it demonstrated the necessity of limiting the current density to a reasonable 30 ma/cm<sup>2</sup>.

Experiment #14d was a full 2<sup>3</sup> factorial which varied the primary factors (i. e., separator thickness, amount of cathode water, and discharge rate) influencing, at that point, the moderate temperature, high discharge region being investigated. The yields of these cells were substantially improved above the 2.0-volt level and the Yates analysis further supported initial indications of preferred levels (i. e., thinner separators, higher percentage of cathode water, and lower discharge rate) of the primary factors. Much of the improvement is attributed to limiting the current density to 30 ma/cm<sup>2</sup>. The mean-effect data (regression equation coefficients for ampere-hours/lb of CuF<sub>2</sub> to the 2.0-volt level are as follows:

Experiment #14d - Mean-Effect Data

Factors	Mean-Effect Data
A - Discharge Rate	-13.8
B - Separator Thickness	-11.8
AC - Interaction	+11.8
C - Cathode H <sub>2</sub> O Content	+11.3
ABC - Interaction	+ 4.3
BC - Interaction	- 2.3
AB - Interaction	+ 1.8

The three primary factors and one interaction were relatively significant. The interaction AC can be interpreted as follows:

		Cathode H <sub>2</sub> O Content	
		2%	8%
Discharge Rate	20	176	175
	30	150	173

As the chart indicates, the level of water only becomes important to sustain discharge at the higher rate. At a lower discharge rate the amount of water is unimportant. Discharge data for Experiment #14d can be found on Table 23. Figures 27 through 30 illustrate the discharge profile.

Based on the results obtained in Experiments #12 and #14, it appeared that the optimum design reached in the high temperature, low discharge rate subregion (Experiment #6 - Step 4) will not perform at the moderate temperature, high discharge subregion.

#### 3.2.2.3.4 Experiment #15

Returning to the format used in Experiment #12, values of the factor levels were changed to those shown in Table 24 to reflect the knowledge gained from Experiment #14.

TABLE 24  
Experiment #15 Factors and Levels

	(S <sup>-</sup> )	L	<u>Levels</u> C. Pt.	H	(S <sup>+</sup> )
Separator Thickness	.010	.015	.020	.025	.030
Electrolyte Molarity	2.1 M	2.3 M	2.5 M	2.7 M	2.9 M
Cathode Thickness	.050	.060	.070	.080	.090
Cathode H <sub>2</sub> O Content	6%	8%	10%	12%	14%
Cathode Conductor Content	7%	8%	9%	10%	11%

The yield data obtained from Experiment #15 were subjected to a Yates analysis of mean-effect levels which revealed a distinct preference for the lower molarity electrolyte, the .060" cathode, and the .015" separator. Table 25 lists the results for the values obtained on the basis of ampere-hours/lb of CuF<sub>2</sub>. Table 26 describe the construction details and Table 27 lists the discharge data for Experiment #15. Figures 31, 32, and 33 provide representative performance curves. As can be seen in Table 27, the order of significant factors depends on whether the 2.5 or 2.0-volt level criterion is used. For the  $y_1$  values, B and C were by far the most significant factors. Factor A was significantly negative. For the  $y_2$  values, C retained its major significance while A and B became only moderately significant.

The  $y_1$  mean-effect values indicated no interactions of major significance and only a few having moderate significance, the highest of which was the BD combination. The following chart describes the interaction:

		Electrolyte Molarity - B	
		<u>2.3 Molar</u>	<u>2.7 Molar</u>
Cathode H <sub>2</sub> O Content - D	8%	71	17
	12%	58	25

The chart's preference corresponds with the influence of the high mean-effect value of factor B.

The  $y_2$  mean-effect values indicated that only the BC interaction had major significance. The BD interaction which showed importance to the 2.5-volt level became relatively unimportant to the 2.0-volt level. The following chart describes the interaction:

		Electrolyte Molarity - B	
		2.3 Molar	2.7 Molar
Cathode Thickness - C	.060"	155	174
	.080"	152	105

The chart indicates that when the electrolyte is at the 2.3-molar level there is no preference in terms of cathode thickness; however, at the 2.7-molar level there is a definite preference for the thinner cathode thickness.

An interesting result obtained from this experiment is the low significance of the cathode water on performance. This factor, in the past, had repeatedly indicated just the opposite. The cause for these results may be that the levels chosen did not cover a large enough range to reflect on the performance of the cells and were both adequate in sustaining relatively the same output at this temperature and discharge rate. This initially suggested that the water level is important in that it must be at least eight percent; however, interpretation of the results of Experiments #15c and #15d showed that the optimum water level appeared to be between 14 and 17 percent.

Center point tests revealed a definite advantage, at the 10 percent water level, of a glass separator having a double layer over one having a single layer. This can be seen by comparing the two performance curves in Figure 34.

The purpose of Experiment #15b was to investigate extreme departures of single factor levels from the center point values. It treated the star points or the outer limits of factor variation, shown in Table 24A, by placing a single factor at a star point while the other factors were maintained at the center point. For each factor, a set of cells tested the high star point levels and another the low star point levels. In relating the star point cells to the center point cells in terms of  $y_2$  yields (ampere-hours/lb of  $\text{CuF}_2$  to 2.0 volts), only the cells at the high water level (14 percent) performed equivalently to or better than those of the center point. On the basis of  $y_1$  yield (ampere-hours/lb of  $\text{CuF}_2$  to 2.5 volts) many of the star point cells provided better or more consistent values than the center point cells, which reflect the higher voltage levels maintained at some of the star points. For example, the maximum voltages obtained at the low separator star point were 2.81 and 2.80 volts, whereas at the center point the average was only 2.52 volts.

Comparison of the individual star points indicated that each of the five factors was highly significant when the levels for each were displaced to the degree that they were in this experiment: voltage levels were increased with the lower separator thickness (.010"), lower electrolyte molarity (2.1 molar), higher percentage of cathode conductor (11 percent), and the higher level of cathode water (14 percent) while thinner cathodes (.050") contributed to greater efficiency. Cell construction details and discharge data are shown in Tables 28 and 29. Curves of performance of the center point are shown in Figure 35. Examples of star point cell performance are shown in Figures 36 and 37.

From the experimentation involving higher levels of cathode water, there were clear indications of positive effect on discharge efficiency as the water was increased.

This was very evident when comparing levels in the four to six percent range against values greater than 10 percent; however, the benefits begin to diminish

at levels above 14 percent. For the  $+10^{\circ}\text{C}$ ,  $30\text{ ma/cm}^2$  discharge region, the area of maximum response occurred between 14 and 17 percent cathode water. This deduction was made from the interpretations of the results of Experiments #15c and #15d. The results of Experiments #15c and #15d are not completely consistent with each other, but this variation is attributed to either procedural changes or to subtle differences in the constituent materials. Cell construction details and discharge data for Experiment #15c, #15d, and #15e are shown in Tables 30 and 31. Discharge curves comparing cells with 15, 17, and 19 percent of  $\text{H}_2\text{O}$  are shown in Figure 38.

As the separator thickness was decreased in Experiment #15e, the voltage level became maximum at the lowest level treated, .010". However, in terms of efficiency to both the 2.5 and 2.0-volt levels, the highest value was obtained at .015" thickness. A comparison of discharge curves from Experiment #15e is shown in Figure 39.

#### 3.2.2.3.5 Experiment #17

On the basis of calculations from data generated in Experiments #15a and #15b, Experiment #17 treated a vector of predicted steepest response which had the following characteristics:

Separator Thickness	- decreasing .021" to .013" in five increments
Electrolyte Molarity	- decreasing 2.5 to 2.1 in five increments
Cathode Thickness	- increasing .060" to 0.100" in five increments
Cathode Water Content	- increasing 9.5 to 11.5% in five increments
Cathode Conductor Content	- increasing 8.8 to 9.6% in five increments

Experiment #17 cell construction details and discharge data are shown in Tables 32 and 33.

A value of 200 ampere-hours/lb of  $\text{CuF}_2$  was expected to occur at the fourth step in the progression; however, this did not occur as can be seen in the plot of cell yields versus factor combinations shown in Figure 40. On the basis of  $y_2$  yield, the plot was negatively sloped from the first factor combination with a dip occurring at the third level, and a wide range of values found for the fourth and fifth factor levels. Discharge curves representing each level are shown in Figures 41 through 43.

The poor performance of the vector chosen was attributed to the direction one of the factors was varied. The Yates analysis of Experiments #15a and #15b gave a large negative mean-effect for factor C, cathode thickness, for both  $y_1$  &  $y_2$  yields. This means that on the basis of these data, cell performance preferred the thinner cathode. Instead the design of the vector called for increasing the cathode thickness rather than decreasing it from the .060" preferred level.

#### 3.2.2.3.6 Experiment #24

Experiment #24 is another vector determined from an additional analysis of Experiment #15 data by NASA. It has the following characteristics:

Separator Thickness	- increasing .020" to .030" in three increments
Electrolyte Molarity	- increasing 2.5 to 2.7 in three increments
Cathode Thickness	- decreasing .070" to .050" in three increments
Cathode Water Content	- increasing 13.0 to 19.0% in three increments
Cathode Conductor Content	- increasing 9.0 to 11.0% in three increments

Cell construction details and discharge data are shown in Tables 34 and 35.

While performance of the cells decreased for each progressive step of the vector, as shown in Figure 44, the inclusion of a second layer of separator material significantly improved the performance of the No. 3 vector position (see Figure 45).



Considerable variation in the results of the No. 2 and No. 3 vector positions was encountered, suggesting excessive shorting through the separator. A greater incidence of shorting was noted as the cathode water percentage was increased at the higher temperature and with single layer separators.

#### 3.2.2.4 High Temperature, High Discharge Rate Investigations

##### 3.2.2.4.1 Experiment #16

The first investigations into the high temperature, high discharge subregion were undertaken in Experiment #13. Experiment #16 is a continuation of this work and uses the results of Experiment #13 as a point of reference. The two basic purposes of this work were to establish the general area of factor variation in which the best performance may occur and to determine if some of the construction variables that apply at the moderate temperature, high rate region are also applicable in this area.

The cell used in Experiment #13 contained a two-layer, .023" thick separator, two percent styrene cathode binder, and Asbury Ceylon cathode conductor. Since separator thickness proved to be a significant factor during moderate temperature testing, a .020" single layer separator was introduced in Experiment #16a. Experiment #16b retained the .020" single separator but Conductex carbon replaced Asbury carbon on the basis of its superior performance at moderate temperatures. Experiment #16c returned to Asbury carbon but reducing the amount of binder to 0.5 percent in order to determine the influence of the amount of cathode binder on the performance of the cell. Experiment #16d mated 0.5 percent binder with Conductex carbon in the cathode. Finally for Experiment #16e, the cells had low (0.5 percent) binder content, Conductex carbon of the previous group, and separator thickness increased from one layer (.020") to two layers (.023").

In this series of experiments there were no dramatic changes in the results of one set of conditions to the next. A slight superiority in the performance of the cells in Experiment #16e encouraged the use of the thicker double layer separator especially in light of the greater incidence of copper deposition in the separator at the +40°C temperature than previously seen at the +10°C level. The deposition reflects the increased copper solubility at the higher temperature.

Cell construction characteristics are provided in Table 36 while discharge data and performance curves are shown in Table 37 and Figures 46 through 48.

### 3.2.2.5 Performance Criteria

As a practical test of the validity of the techniques used in this program and of the results obtained to date, the NASA Project Manager requested that a series of performance cells be discharged to a prorated specific electrical requirement covering a potential application where the total cell package would be required to discharge at 80 amperes with a total capacity of 150 ampere-hours. Several design iterations were made and these established that the cell package would contain 24 cathodes, each .070" thick and would need to discharge to 60 percent efficiency at an average current density of 20 ma/cm<sup>2</sup> to the 2.0-volt level. The prorated demonstration cell contained a single anode and two cathodes. The cell construction details are shown in Table 38 and discharge data are shown in Table 39.

#### 3.2.2.5.1 Experiment #19

The purpose of Part "A" of Experiment #19 was to set the levels of the three primary construction variables. The ranges utilized were:

- |                   |                  |
|-------------------|------------------|
| 1. electrolyte    | 2.3 to 2.7 molar |
| 2. water          | 4 to 8 percent   |
| 3. carbon content | 7 to 11 percent  |

The ranges chosen for the variables were based on results of previous experiments most closely associated with this discharge rate and temperature. It was quite apparent from the test results that cells operated at  $+10^{\circ}\text{C}$  using 2.7 molar electrolyte, 11 percent carbon, and eight percent water were decidedly better than those operated at other levels. This is shown in Figure 49 and reflected in Figure 50 in terms of cathode efficiency and watt-hours/lb of  $\text{CuF}_2$  to 2.0 volts.

Using the cell configuration providing the best results of Experiment #19a, the test temperature was varied from  $+0$  to  $+40^{\circ}\text{C}$  in Experiment #19b. The cathode efficiency, as shown in Figures 51 and 53, was greatest at  $+40^{\circ}\text{C}$ , and as the temperature decreased below  $+5^{\circ}\text{C}$  the energy factor (watt-hours/lb of  $\text{CuF}_2$  to 2.0 volts) decreased rapidly. Despite the variation in performance that resulted from temperature, none of the cells performed below the 60 percent cell efficiency criterion.

Comparison in the levels of electrolyte molarity in Experiment #19c, as shown in Figures 52 and 53, indicated that the level of this factor was not critical to the performance of the cell. This further suggested that the changes in performance observed in Part "A" of this experiment were due to the variation in cathode water and carbon and not electrolyte molarity.

It is encouraging to note that by the use of parametric information already generated, it was possible to design a cell that wholly met the set of electrical requirements with relative ease.

### 3.2.2.6 Anhydrous Cell Experiments

#### 3.2.2.6.1 Experiments #18b, #20, and #21

The first attempt of this program to investigate the effect on performance of no water in the cathode took place in Experiment #18b. The cells run initially, represented by Cell F-398, showed extremely poor performance. In contrast, an anhydrous cell, with some structural difference, run previously by our Research

Group, showed high performance. Another cell, F-427, incorporating many of those design features was discharged with favorable results. The cell construction details and discharge results are shown in Table 40.

With Cell F-427 as the design basis, Experiment #20 was carried out changing the level of one factor per test group to determine which design parameters were most critical to anhydrous cell performance. The individual factors tested were cathode conductor content and electrolyte molarity. Individually the highest performance in terms of ampere-hours and watt-hours per pound of  $\text{CuF}_2$  were obtained at the highest carbon content level (21%), at an electrolyte molarity of between 2.4 and 2.7 molar, and in a two-plate cell configuration. Cell construction details and discharge data are shown in Tables 41 and 42. Curves portraying the effects of carbon concentration and electrolyte molarity on cathode efficiency are shown in Figures 54 and 55.

Combining the maximum performance levels of each of the tests in Experiment #20, Experiment #21 investigated the effects on immediate discharge performance of separator material and the presence of an  $\text{SO}_2$  atmosphere. Construction details and discharge data are shown in Tables 43 and 44. In Part "B" of Experiment #21, energy figures improved to a consistent 600 watt-hours/lb of  $\text{CuF}_2$ , the voltage levels were among the highest obtained for cells tested to date for a discharge rate of 10 ma/cm<sup>2</sup>, and efficiencies ( $\eta$ , ampere-hours/lb of  $\text{CuF}_2$ ) were slightly higher than those obtained with the individual maximum design levels tested in Experiment #20.

Experiment #21 proved that a Honeywell separator (Part #2974), identified by internal research as a potential means of improving cell performance, had a negative effect on immediate discharge performance and in combination with five psi pressure of  $\text{SO}_2$  gas, the effect was compounded. When  $\text{SO}_2$  was used with a standard separator the results obtained were essentially the same as those of Experiment #21b for efficiency.  $\text{SO}_2$  was used because it had been identified as a possible means for improving active stand capability. A comparison, in terms of cathode efficiency, of the four parts of Experiment #21 are shown in Figure 56.

### 3.2.2.6.2 Experiments #22, #23, and #26

Experiment #22 continued the investigation into possible active stand materials by establishing the performance and discharge characteristics of cells using  $\text{LiBF}_4$  electrolyte salt in comparison to cells of Experiments #21b and #21c, i.e., with and without  $\text{SO}_2$ . Cell construction details and discharge data of Experiments #22 and #23 are shown in Tables 45 and 46.  $\text{LiBF}_4$  appeared to reduce the voltage level somewhat. The use of  $\text{LiBF}_4$  electrolyte salt with  $\text{SO}_2$  at the 2.5-molar level provided essentially the same performance as a cell using 2.5-molar  $\text{LiAsF}_6$  without  $\text{SO}_2$ . Interestingly,  $\text{SO}_2$ , when used with  $\text{LiBF}_4$ , caused an extremely rapid voltage decay at the end of the discharge as shown in Figure 57. In the absence of  $\text{SO}_2$ , the voltage decayed at a gradual rate during the last 50 percent of the discharge period (see Figure 58).

For reasons of economy and long term chamber seal integrity, the test chamber was reduced in size to accommodate a 1-1/2" x 5/8" cell and had the form of a modified glass compatibility tube. Such a chamber is illustrated in Figures 6 and 7. Cells could be accommodated in either the flooded or wicked mode. The tops of the tubes were fitted with the necessary hardware to provide pressure monitoring, electrolyte entry, and sealing.

In order to develop a background of comparative discharge data, Experiment #23 was conducted in the new test chambers but otherwise under the same conditions as in Experiment #21b. There was little or no loss from test chamber influences when testing was carried out in the wick mode with the glass pressure tubes; however, in contrast, there was approximately a 10 to 15 percent loss when cells were run in the flooded mode in the same glass tubes. This comparison can be seen in Figure 59.

Performance characterization of the new standard anhydrous cell (based on results of Experiment #21b) in terms of discharge rate and temperature was carried out in Experiment #26 in 2" x 3" cells. The results of Experiment #26a indicated that performance began to drop off as the discharge rate exceeded 20 ma/cm<sup>2</sup>. At the

20 ma/cm<sup>2</sup> discharge rate the efficiency was equal to that of the 10 ma/cm<sup>2</sup> rate but the voltage was slightly lower. The voltage profiles for these cells are given in Figure 60.

The anhydrous cell is extremely sensitive to the temperature at which it is discharged. There was essentially equal performance at +35 and +40°C at 10 ma/cm<sup>2</sup>, but at +30°C the yields dropped significantly to less than 30 percent efficiency. The profiles for these Experiment #26b cells are given in Figure 61.

Dramatic improvements were made in the performance of these cells at +30°C by the substitution of SO<sub>2</sub> gas for argon in the cell system. In Figure 62 the average discharge voltage to a 2.0-volt cutoff of Experiment #26c is compared to the results of Experiment #26b (without SO<sub>2</sub>). Voltage was increased by 10 percent, the discharge life was increased by a factor of eight, the efficiency was increased by a factor of six, and the energy was increased by a factor of seven.

With SO<sub>2</sub> present it is expected that efficient discharge at still lower temperatures is possible but tests to determine low temperature behavior with SO<sub>2</sub> were not performed.

The construction characteristics and discharge data for all cells in Experiment #26 are given in Tables 47 and 48, respectively.

### 3.2.2.7 Active Stand Experiments

During the month of February 1971 the contract was amended to remove the Task 3 requirement of 20 demonstration units and replace it with work directed toward the study and identification of system capabilities in the active stand area. Active stand capability is recognized as an important requirement in the performing ability of the system if it is to find use in potential applications.

#### 3.2.2.7.1 Experiments #18a and #18c

The separator is a particularly important component in active stand cells. Selection of an optimum separator was made prior to actual active stand testing. Three separator thicknesses at two temperatures were investigated in Experiment #18a, and a porvic separator material, recommended by our Research Group, was tried in Experiment #18c. The construction details and discharge data can be found in Tables 49 and 50, respectively.

Performance, as seen in Figure 63 at the lower temperature (+10°C), was not significantly affected by the three separator levels used. However, at the higher temperature (+40°C), as seen in Figure 64, the maximum level of performance was achieved when the thickest combination, two layers of glass totaling .020", was used. One of the significant causes of this difference was the prevalence of the deposition of copper in the separator material at high temperature testing. It was apparent that the additional separator material in the two-layer cells provided sufficient protection to prevent dendritic shorting between the electrodes and thus enabled the cell to discharge more efficiently. It is also noted that the higher temperature cells showed no evidence of the initial low voltage interval that occurred in the lower temperature cells.

Through Experiment #18c it was concluded that porvic material, a porous membrane-type of separator manufactured by Porvair Ltd. of England, cannot be completely substituted for glass but that one of the layers in a two-layer glass system could be replaced by porvic with a slight lowering of the average voltage level (see Figure 65). However, it was shown in Experiment #25c that porvic inhibits wicking of the separator and, therefore, it was discarded from further consideration in active stand investigation.

#### 3.2.2.7.2 Experiment #25

Based on the recommendations and experience of the Honeywell Power Sources Center Research Group, SO<sub>2</sub> gas, an additive which seems to reduce the solubility

and migration of  $\text{CuF}_2$ , was introduced to endeavor to improve the active stand characteristics of this system.

The purpose of Experiment #25 was to establish the seven-day active life characteristics of several types of cells, including the standard anhydrous cells (from Experiment #21b), with and without  $\text{SO}_2$ , in both the 1-1/2" x 5/8" and 2" x 3" sizes.

In the first tests of Experiment #25a, inadequate wicking of cells, as shown by slow activation in the immediate discharge mode, was discovered. This was remedied by increasing the number of wicks per cell. The flooded condition, normally existing in the standard NASA fixture, was found to be detrimental to active stand performance.

This phenomenon was further explored in Experiments #28 and #29 where it was found that for optimum cell performance the quantity of electrolyte is critical.

The influence of  $\text{SO}_2$  on cells discharged after seven days of active stand was dramatic. The cathode efficiencies of these cells without  $\text{SO}_2$  were only 7 to 16 percent and voltage levels were consistently below 2.5 volts. With  $\text{SO}_2$  included, the voltage levels remained high and the efficiencies ranged from 69 to 80 percent. Discharge curves illustrating the effect of  $\text{SO}_2$  are shown in Figures 66 and 67.

When a layer of porvic was placed between two glass separator layers in Experiment #25c, the porvic inhibited otherwise normal wicking. In the immediate discharge mode the cells with porvic had an efficiency of 25 to 30 percent, while after seven days of active stand the efficiency rose to 65 percent with porvic (see Figure 68).



After this initial series of active stand tests, one cell (G-558) exhibited an extremely corroded anode lead. This was discovered as a result of a post-test analysis to determine the cause of an inefficient discharge. Degrading of silver anode leads had been noticed in many active stand tests. Experiment #27 was generated to further investigate this phenomenon.

The construction characteristics of the cells in Experiment #25 are shown in Table 51 and the resulting data are shown in Table 52.

### 3.2.2.7.3 Experiments #27, #28, #29, and #31

As a result of corrosion of the silver anode leads, an examination was made of the effect of several anode grid materials on discharge performance after 7 and 14 days active stand with an  $\text{SO}_2$  atmosphere. Eighteen cells were fabricated in Experiment #27, six each containing Type 304 stainless steel, nickel, and copper grid materials.

After seven days of active stand, the efficiencies of all the cells were about the same (see Figure 69). The voltage level for the copper units was the highest which probably reflects the relative electrical conductivity of the metal. None of the three materials exhibited the seven-day corrosion problems of the silver grid. Only a slight surface discoloration of the nickel and stainless steel materials was noticeable. After 14 days of active life, however, the discharge performance of the cells with copper grids was significantly better than the values for the nickel and stainless steel cells. A comparison of the best performing cell of each type can be seen in Figure 70. Experiment #27 has empirically established that a relationship exists between the grid material and cell performance. Since copper, as a grid material, resulted in satisfactory and more consistent cell performance after the active stand interval, it replaced

silver as the standard grid material in the anode for subsequent test cell. Cell construction details and discharge data for Experiment #27 are shown in Tables 53 and 54.

In Experiment #28, 2" x 3" cells, using limited quantities of electrolyte (15 and 20 cc), and employing an SO<sub>2</sub> atmosphere, were discharged immediately after activation. Performance decreased with a decrease in electrolyte volume as shown in Figure 71. Cell construction details and discharge data are shown in Tables 55 and 56.

In Experiment #29 slightly greater quantities of electrolyte (20 and 25 cc) were introduced to the cells and their performance were compared. Upon immediate discharge, cells of both volumes produced almost identically high discharge efficiencies (see Figure 72). After 7 and 14 days of active stand, only the 20 cc cell produced a high efficiency discharge (see Figures 73 and 74). Cell construction details and discharge data are shown in Tables 57 and 58.

Of major importance in this series of tests is the attainment, with the use of SO<sub>2</sub> gas, of the same cathodic efficiencies after 7 and 14 days of active stand, +40°C storage, as obtained upon immediate discharge. The cells of Experiment #29 produced the best capacity retention results of the work done under the contract.

The objective of Experiment #31 was to determine the discharge characteristics of a group of cells in which the CuF<sub>2</sub> and the electrolyte used were obtained from different sources.

The standard CuF<sub>2</sub> material, obtained from Ozark-Mahoning, used in all previous testing, was compared to Battelle-Northwest CuF<sub>2</sub> with a stated purity of 99.99 percent.

Electrolyte was made using  $\text{LiAsF}_6$  from three sources:

1. Honeywell                      Made according to standard electrolyte preparation procedure PSC 8015-1 which used, as starting materials, Ozark-Mahoning  $\text{KAsF}_6$  and Foote Mineral  $\text{LiBF}_4$ .
2. U. S. Steel                      Prepared directly by dissolving their  $\text{LiAsF}_6$  in distilled spectroquality MF obtained from Matheson, Coleman & Bell. The salt sample gave a basic ( $\text{pH} = 11.0$  at  $1\text{M}$ ) water solution and has appeared to be stable in MF solutions for periods extending past two weeks.
3. Midwest Research Institute (MRI)                      Prepared directly by dissolving MRI  $\text{LiAsF}_6$  in distilled spectroquality MF obtained from MCB. This resulted in an acid ( $\text{pH} = 3.0$  at  $1\text{M}$ ) aqueous solution. The material was made under NASA Contract No. NAS 3-12979 by MRI.

Except for combinations previously tested, the experimental test plan incorporated all combinations of the above and is shown in Table 59. The results of the tests are shown in Table 60 and discharge curves of these results are shown in Figures 75 to 77.

In summary, both Ozark-Mahoning and Battelle  $\text{CuF}_2$  provided the most efficient discharges at all levels of storage when used with Honeywell prepared electrolyte. The discharge decay rate was approximately 15 to 20 percent per week. The discharge decay rate for MRI electrolyte was approximately 20 to 30 percent per week. The U. S. Steel electrolyte, while comparable with Honeywell units in the zero storage time mode, deteriorated almost entirely after seven days regardless of the  $\text{CuF}_2$  source.

#### 3.2.2.7.4 Experiments #30, #32, and #33

Optimization of the amount of  $\text{SO}_2$  gas in small standard cells to provide maximum active stand performance was the subject of Experiment #30. Cells were discharged at two  $\text{SO}_2$  quantity levels (.090 and 0.135 g/g electrolyte) and at two storage temperature levels (+20 and +40°C). Inconsistent and poor results as seen in Figure 78 for factor combinations prejudged to provide satisfactory performance, led to an investigation of the effect of electrolyte age (Experiment #32), and a further study of minimum  $\text{SO}_2$  quantities (Experiment #33) for satisfactory active stand performance. Experiment #30 cell construction details and discharge results are shown in Tables 61 and 62.

Experiment #33 explored the effect on active stand cell performance of low levels of  $\text{SO}_2$  gas. Previous results (Experiments #25a, #27, and #31) with  $\text{SO}_2$  showed that best performance occurred at the 0.113 g/g of electrolyte level but the relative importance of this level was not known. This was confirmed by the marginal behavior of the .090 g  $\text{SO}_2$  level and by the degradation of performance at the .023 and .045 g  $\text{SO}_2$  levels. Cell construction details and discharge results are shown in Tables 63 and 64. A performance curve for the .090 g  $\text{SO}_2$  level is shown in Figure 79.

The objective of Experiment #32 was to learn if the age of electrolyte had an affect on active stand cell performance. The test results showed inconsistent behavior within all groupings. However, the most efficient cell used the oldest electrolyte, which seemed to refute a former tentative conclusion that electrolyte age had a negative effect on performance. A moderate correlation between post-test weight loss of cell chamber assemblies and poor performance revealed the possibility of an  $\text{SO}_2$  gas leakage. Since Experiment #33 established that a minimum quantity of 0.113 g/g electrolyte must be maintained in a cell of this size, any  $\text{SO}_2$  loss would have a detrimental effect on the cell's active stand performance. Unfortunately, this program was concluded before this supposition could be confirmed. Cell

construction details and discharge results are shown in Tables 65 and 66. A performance curve of a cell containing the oldest electrolyte is shown in Figure 80.

#### 3.2.2.8 Other Experiments (Experiments #5 and #10)

The purpose of Experiment #5 was to provide an evaluation, in terms of cell performance, of two separator materials, dacron and rayon polypropylene, that were selected on the basis of the second series of compatibility tests discussed in 2.2.1.

The results of the experiment were not appreciably improved as compared to previous tests, but the test supplied sufficient data to select the best of the two materials. The Yates analysis of mean-effect response indicated separator material as a significant factor, with the rayon polypropylene being preferred. Separator thickness and temperature were not significant influences for this experiment, but discharge rate, although not as prominent as the material, did indicate a preference for the lower level. The construction details for this experiment are shown on Table 67, the discharge data on Table 68, and the mean-effect data on Table 69.

Of particular interest in this experiment was the unusually high significance of one of the interactions, the AB/CD combination. In the structure of the experiment the substitution of factor D for treatment combination ABC had the effect of also confounding the AB (discharge rate - temperature) interaction with CD (separator material - separator thickness) interaction. The cause of the high mean-effect may have been due to one or both of the interactions involved.

To illustrate the interaction of the factors involved, the following charts were prepared:

Interaction AB

		Temperature	
		+31°C	+43°C
Discharge Rate	13 ma/cm <sup>2</sup>	157.0	95.9
	19 ma/cm <sup>2</sup>	87.1	134.0

The numbers listed are the averages of the two ampere-hours/lb of CuF<sub>2</sub> responses for the factor levels indicated. Reference to this treatment of interactions can be found in the Design and Analysis of Industrial Experiments, by Davies, on page 255 and the example given on page 456.

In the above chart, the indication is that temperature discharge rates do interact, and that the performance is best when the factors are either both at the high level or both at the low level.

The other factor interaction of the confounded pair is the CD combination.

Interaction CD

		Separator Material	
		Dacron	Rayon Polypropylene
Separator Thickness	.005"	125.0	116.0
	.010"	67.1	166.0

The above chart indicates that at the .005" thickness there is little distinction between the two materials as to their effect on performance; however, at the .010" thickness there is a definite advantage with the rayon polypropylene material.

Both of the interactions indicated by the charts are logical in their occurrence, and the total value of the mean-effect is considered to be the result of both.

Experiment #10 was a short, preliminary study of the effect of a dual separator system (different materials in one cell) on cell performance. The cells involved were identical in construction, with the exception that one used all glass separators and the other used a combination of glass and rayon polypropylene. The yields obtained from both types of cells were essentially equivalent, which is significant because it implies that rayon polypropylene, with its inherent advantages, can be used without deterring cell performance. These advantages include heat sealability (important in the construction of larger number of cells) and minimum thickness, which reduces electrolyte requirements and heightens the possibility of high discharge capability. Figure 81 reflects the discharge curves of these cells.

### 3.3 Analysis of Cell Discharge Gases

One of the additional responsibilities of Task 2 was the measurement of the rate at which heat and pressure generate during cell discharge. These measurements are reflected on many of the discharge curves that are included in this report. It is also required that the composition of the gases responsible for the pressure buildup be determined.

The composition of the gases and vapors was determined through the use of gas chromatography. Sampling of the gases was done when discharge voltage reached its minimum limit (1.9 volts).

To gain as complete a composition spectrum as possible it was necessary to use two different columns in the GC. A 5A molecular sieve unit established hydrogen,  $O_2$ -Ar, nitrogen, methane, and carbon monoxide. A chromosorb 102 unit provided estimation on the quantity of air,  $CO_2$ ,  $H_2O$ , methane, and methyl formate. Table 70 shows the typical values for some of the gases present in cells tested.

It should be noted that the data shown in Table 70 does not reflect the large amounts of CO that were being detected before our sampling procedure was standardized to the point where the discharge voltage reaches the minimum level. The initial cells,

analyzed primarily for equipment procedures, had been standing in the activated open circuit condition for several hours after discharge, and the indicated large amounts of CO. The implication is that the presence of CO is related to these conditions just described.



#### 4.0 Summary of Results

Performance characteristics of the electrochemical system lithium/methyl formate-lithium hexafluoroarsenate/copper fluoride have been investigated under the terms of this contract, and regions of optimum performance as a function of cell design and operational variables have been identified.

The first task, the evaluation of candidate separator materials in terms of electrolyte compatibility and electrical resistance, identified three favorable materials. Subsequent use in Task 2 testing proved the best material was glass fiber mat grade 934H manufactured by Reeve Angel. The thickness and number of layers for best cell performance was dependent upon the discharge rate and temperature of the cell. As the rate increased the deposition of copper upon the separator increased, thereby requiring a thicker separator to prevent shorting by the dendritic growth. On the other hand, under conditions of low discharge rate, it was necessary to decrease internal cell resistance by minimizing the separator thickness. Discharge temperature also influenced dendritic growth. At high temperature levels there was a tendency for more copper deposition in the separator.

Most influential of the 15 prescribed factors, upon the performance of the test cells in Task 2, were operating temperature and discharge rate. The best yield of the program, in terms of ampere-hours/lb of  $\text{CuF}_2$ , was provided by an anhydrous system at the  $+40^\circ\text{C}$  level. However, the anhydrous cell was extremely sensitive to its discharge temperature. Its yield dropped dramatically from 85 percent efficiency at  $+40^\circ\text{C}$ , to less than 30 percent at  $+30^\circ\text{C}$ . "Wet" (i. e., containing a finite percentage of copper fluoride dihydrate) cathode cells also performed well at  $+40^\circ\text{C}$  but the decline in efficiency with a decrease in temperature was not as severe. Interestingly, the addition of  $\text{SO}_2$  gas to the anhydrous cell system induced impressive performance improvement at  $+30^\circ\text{C}$ , increasing the cathode efficiency from less than 30 to 70 percent.

A maximum practical cell discharge rate of  $30 \text{ ma/cm}^2$  was established in this program.

The level of water in "wet" cathodes affected the performance of these cells at both high and low discharge rates. At  $30 \text{ ma/cm}^2$ , optimum performance was attained with cells containing cathode water at the level of 14 to 17 percent of the cathode weight. At  $9 \text{ ma/cm}^2$ , optimum performance was reached with a water content of four percent. While the level of cathode water did not strongly affect the performance of the cell at lower discharge rates, the water level did become important in sustaining discharge at the higher rates.

Adjustment of other factor levels was not as critical to the performance of these cells. Minor level adjustments of the electrolyte concentration, cathode thickness, cathode conductor material and content, cathode binder content, and the electrolyte volume influenced the performance of the cells somewhat at each discharge condition.

The condition of active stand life was also examined in this program. The ability of a cell to discharge efficiently after 7 and 14-day active stand was clearly improved by the addition of  $\text{SO}_2$  gas to the cell. In fact, active stand was achieved only through the use of  $\text{SO}_2$ . Once active stand capability was attained, electrolyte volume and anode grid material factors affected cell performance. Active stand life was negatively affected by the condition of electrolyte flooding since such a condition induced internal cell self-discharge. When the amount of electrolyte was correctly adjusted, there was essentially no loss after 14 days of active stand at  $+40^\circ\text{C}$  storage. Because silver anode grid material deteriorated after 24 hours in active stand and interfered with cell performance after seven days, the silver material was replaced by copper.

It became clear that the optimum design reached for one temperature-discharge rate subregion would not be the optimum design for another temperature-discharge

rate subregion. The battery must be customized to the subregion or condition most likely to be encountered.

The use of statistical disciplines concerning factorial and fractional factorial designs were very useful in arriving near the optimum level quickly and with minimum testing. However, as the optimum performance level was approached, the statistical methods were not directional enough and were therefore replaced by the more classical direct comparison experimental methods.

Anhydrous cell technology investigations produced the highest performance results of the program. Obtained were consistent yields of 200 ampere-hours/lb of  $\text{CuF}_2$  and 600 watt-hours/lb of  $\text{CuF}_2$  at 10 ma/cm<sup>2</sup> and +40°C.

TABLE I

Compatibility Data Summary of Separator Materials in 3.0-Molar LiAsF<sub>6</sub>/MF - Initial Series

Material	<u>Visible Changes</u>				<u>Percentage Weight Change</u>				<u>Percent Change Thickness Uncompressed</u>			
	Scan 1	Scan 2	Scan 3	Scan 4	Scan 1	Scan 2	Scan 3	Scan 4	Scan 1	Scan 2	Scan 3	Scan 4
Glass	1	1	1	1								
Pellon	2	2	2	2	-1.1	- 0.6	0	-0.9	- 8.0	0	0	+ 8.3
Porvic	3	3	3	3, 4	-1.9	+ 0.2	+16.3	-4.1	-20.0	-36.0	-45.5	0
Dacron	2	2	2	3	-1.5	- 1.2	- 3.5	+0.8	+ 3.0	+ 7.0	- 5.0	-14.3
Rayon	2	2	2	2	+4.5	- 2.4	- 2.5	+1.7	+ 5.0	+ 4.0	- 3.6	-14.3
Tyvek	4	5	5	2	+1.4	+ 2.1	+ 5.6	+3.6	+28.0	+25.0	0	0
Synpor		3	3, 5	3, 5		-13.4	- 1.4	-3.5		-41.0	-29.4	0
Latex Glass		1, 6	1, 6	1, 6								

Material	<u>Percentage Width Change</u>				<u>Percentage Length Change</u>				<u>Percent Change Thickness Compressed</u>			
	Scan 1	Scan 2	Scan 3	Scan 4	Scan 1	Scan 2	Scan 3	Scan 4	Scan 1	Scan 2	Scan 3	Scan 4
Glass												
Pellon	-0.3	-0.9	+0.9	-1.4	0	+ 0.5	- 0.3	-8.0	+ 8.0	0	+ 8.0	+33.0
Porvic	+8.5	+9.0	+1.1	+8.6	+6.7	+ 7.0	+ 9.7	+5.9	-35.0	-41.0	-43.8	-25.0
Dacron	+1.1	+1.0	0	-0.4	-1.3	- 0.2	- 1.6	-1.1	-13.0	+21.0	+10.4	+17.0
Rayon	+1.4	-1.0	-0.5	+4.1	+0.7	- 0.6	- 0.6	-0.2	-11.4	+26.0	+12.9	+18.8
Tyvek	0	-0.2	+0.6	-1.4	-0.3	- 0.2	- 0.5	-0.5	- 9.0	0	+12.5	+20.0
Synpor		-3.0	+3.1	-9.7		+ 4.0	+10.1	+5.1		-42.0	-28.6	-14.3
Latex Glass												

<sup>1</sup> Structureless<sup>2</sup> None<sup>3</sup> Edge distorted<sup>4</sup> Electrolyte darker<sup>5</sup> Electrolyte slightly darker<sup>6</sup> Electrolyte much darker

TABLE 2

Compatibility Data Summary of Separator Materials in 3.0-Molar  
LiAsF<sub>6</sub>/MF - Second Series

Electrolyte NASA #13  
 Initial H<sub>2</sub>O - 116 ppm

Material	24 Hrs	<u>Visual Test Observations</u>				% $\Delta$ Weight	% $\Delta$ Length	% $\Delta$ Width	% $\Delta$ Thickness Uncompressed	% $\Delta$ Thickness Compressed
		48 Hrs	72 Hrs	96 Hrs	100 Hrs					
49	1	*	2	3	3	-0.6/-0.7	-0.6/+4.2	-2.7/-2.1	0/0	0/0
69	1	*	2	2	2	+1.2/-0.4	-0.6/-0.6	-1.3/-2.0	0/0	0/0
137	1	*	2	2	2	+2.0/+1.9	+1.1/-0.6	-0.7/-0.7	0/0	0/0
160	1	*	2	2	2	+1.4/+1.6	-1.2/+1.8	-5.3/-2.7	0/0	0/0
181	1	*	2	2	2	+2.0/+1.6	-3.4/-2.2	-1.7/-2.6	0/0	25/25
Control	1	*	2	2	2	-	-	-	-	-

Note: Post-test observations on all materials--no visible physical change.

Two samples of each material were tested.

Material	<u>Tear Strength</u>	
	<u>Before Scan</u>	<u>After Scan</u>
49	2.5 lbs.	7.75 lbs.
69	>10	>10
137	>10	>10
160	>10	>10
181	>10	>10

Material

49 Rayon H465  
 69 Rayon Polypropylene Carded E4208  
 137 Dacron E1482  
 160 Dacron E1486  
 181 Random Polyester H711

<sup>1</sup> No apparent change.  
<sup>2</sup> Electrolyte slightly darker.  
<sup>3</sup> Electrolyte much darker.  
 \* Reading not taken.

TABLE 3A

Separator Material Resistance in 3.0-Molar LiAsF<sub>6</sub>/MF - Initial Series

Material Identification	<u>Single Thickness</u>		<u>Double Thickness</u>		<u>Triple Thickness</u>		<u>Quadruple Thickness</u>	
	Temp.	Resistance Due to Separator	Temp.	Resistance Due to Separator	Temp.	Resistance Due to Separator	Temp.	Resistance Due to Separator
Glass Mat	28.5°C	0.1 $\Omega$	28.6°C	0.1 $\Omega$	28.1°C	1.8 $\Omega$	28.3°C	2.9 $\Omega$
Pellon 2140	29.0°C	0.5 $\Omega$	29.0°C	0.9 $\Omega$	29.9°C	1.1 $\Omega$	28.8°C	1.7 $\Omega$
Porvic	29.2°C	0.6 $\Omega$	28.4°C	0.8 $\Omega$	28.8°C	1.0 $\Omega$	30.2°C	2.7 $\Omega$
Weblox Dacron	28.5°C	0.2 $\Omega$	28.5°C	0.5 $\Omega$	29.5°C	1.2 $\Omega$	28.9°C	2.6 $\Omega$
Weblox Rayon	28.6°C	0.1 $\Omega$	28.9°C	0.3 $\Omega$	28.4°C	0.5 $\Omega$	28.5°C	0.9 $\Omega$
Tyvek 1458	28.9°C	2.0 $\Omega$	29.5°C	4.9 $\Omega$	28.5°C	9.3 $\Omega$	29.2°C	15.9 $\Omega$

<u>Single Thickness</u>	<u>Compressed</u>	<u>Uncompressed</u>
Glass Mat	.010"	.035"
Pellon 2140	.006"	.012"
Porvic	.008"	.011"
Weblox Dacron	.029"	.140"
Weblox Rayon	.032"	.140"
Tyvek 1458	.005"	.008"

TABLE 3B

Separator Resistance in 3.0-Molar LiAsF<sub>6</sub>/MF - Second Series

PSC I.D. No.	Description	Thickness Uncompressed	Thickness Compressed	Resistance * in Ohms Single Layer	Resistance * in Ohms Double Layer
32	Aclar 33C	.0022"	.0019"	60.4	93.2
83	Carded Rayon H773	.006"	.005"	5.4	9.5
155	Dacron Polyester M1480	.004"	.0035"	3.7	6.1
70	Rayon H635	.003"	.0025"	2.9	5.1
178	Celanese Polyester EM474	.0055"	.004"	2.2	4.2
180	Carded Rayon E4212	.0035"	.003"	1.4	2.6
177	Dacron EM415	.003"	.0025"	1.1	2.1
137	Dacron E1482	.006"	.005"	1.0	1.6
179	Polypropylene E1451	.006"	.004"	0.8	1.2
181	Random Polyester H711	.005"	.0045"	0.7	1.2
182	Rayon Polypropylene Carded 50/50 E4103	.008"	.005"	0.5	0.8
160	Dacron E1486	.005"	.0035"	0.5	0.8
69	Rayon Polypropylene Carded E4208	.011"	.005"	0.5	0.7
48	Rayon E8305	.080"	.022"	0.3	0.3
49	Rayon H465	.042"	.011"	0.2	0.3

\* Temperature Range: 29.5°C to 30.0°C

TABLE 3C

Compatibility Scan - Candidate Plastic Case Materials

Material	24 Hrs	48 Hrs	Observations			168 Hrs	% $\Delta$	% $\Delta$	% $\Delta$	% $\Delta$	Post-Test Observations on Sample
			96 Hrs	120 Hrs	Weight		Width	Thickness	Length		
<u>+30°C</u>											
Polyphenylene Oxide	1	1	1		1	+1.0	0	0	0	Slight darkening of surface color.	
Polysulfone	1	1	1		1	+1.7	0	0	0	Very slight surface etching.	
Glass Filled Polysulfone	1	1	1		1	+0.9	0	0	0	No apparent change.	
Styrene	1		1	5		+2.7	+1.5	+ 2.4	+0.4	Sample edges rounded, slight bulge in center.	
Hi Impact Styrene	1		1	5		+7.1	0	+15.8	+0.4	Same as styrene.	
Polycarbonate	1		1	5, 6		+7.4	+2.6	+ 8.6	+0.3	Surface 40% opaque, edges rounded.	
<u>+60°C</u>											
Polyphenylene Oxide	1	1	3		4	+2.8	+0.4	+ 1.8	0	Surface color much darker.	
Polysulfone	1	1	3		4	+3.9	0	0	0	Etching of surface.	
Glass Filled Polysulfone	2	1	3		4	+3.1	0	+ 2.4	0	Slight lightening of surface color.	
G-10 Glass Fill	1		1	3		+7.0	0.4	+10.8	0	Color of sample 5-10% lighter.	
Polyester	1		1	1							
Polymeric Polyester #2						+1.9	0	0	0	Sample 5-10% darker.	

<sup>1</sup> No apparent change.<sup>2</sup> Electrolyte slightly darker.<sup>3</sup> Slight sediment formation.<sup>4</sup> Electrolyte very dark, near opaque.<sup>5</sup> Sample edges rounded slightly.<sup>6</sup> Sample color change.



TABLE 3D

Compatibility Scan - Candidate Plastic Case Materials

Material	24 Hrs	Observations			% Δ	% Δ	% Δ	% Δ	Post-Test Observations on Sample
		48 Hrs	96 Hrs	120 Hrs	Weight	Width	Thickness	Length	
ABS	1	4	8	8	8.0	8.0	8.0	8.0	Heavy chemical attack, partial dissolving.
Styrene	2	10	5	10	+12.4	8.0	8.0	8.0	Large amount physical distortion.
Hi Impact Styrene	2	10	10	10	8.0	8.0	8.0	8.0	Large amount physical distortion.
Acrylic	1	4	8	8	8.0	8.0	8.0	8.0	Heavy chemical attack, partial dissolving.
Glass Reinforced Polyester	9	5	10	10	+ 1.2	0	0	0	Color lighter than original.
Hi Density Polyethylene	9	10	10	10	+ 1.1	0	+ 1.0	0	No visible change.
Polycarbonate	3	10	10	10	+10.1	0	+14.2	-0.8	Highly crazed surface, color change clear to opaque.
Polypropylene	9	10	10	10	+ 2.0	+1.0	+ 1.4	+0.4	No visible change.
Phenolic	9	6	10	7	- 0.2	0	0	0	Color slightly darker than original.

Observations:

- <sup>1</sup> Complete collapse of sample structure.
- <sup>2</sup> Edges rounded, apparent material softening.
- <sup>3</sup> Sample changed from clear to opaque.
- <sup>4</sup> Heavy apparent chemical attack of sample.
- <sup>5</sup> Slight electrolyte discoloration.
- <sup>6</sup> Moderate electrolyte discoloration.
- <sup>7</sup> Electrolyte very discolored (nearly opaque).
- <sup>8</sup> Not available.
- <sup>9</sup> No apparent change.
- <sup>10</sup> No change from previous observation.

TABLE 4

Experiments #1 and #2 - Mean-Effect Data

Factor	y <sub>2</sub> - Amp-Hrs/Lb CuF <sub>2</sub> to 2.0 v		y <sub>4</sub> - Watt-Hrs/Lb to 2.0 v		y <sub>6</sub> - Watt-Hrs/Cm <sup>3</sup> to 2.0 v	
	Exp. 1	Exp. 2	Exp. 1	Exp. 2	Exp. 1	Exp. 2
A	+32.8	+24.5	+17.3	+15.6	+10.6	+ 8.9
B	-64.1	-43.1	+35.7	-14.3	-22.3	-10.8
C	-16.5	-72.0	-12.0	-32.8	- 7.9	-22.7
D	+23.2	-30.7	+ 1.7	-15.2	- 2.3	-15.3
E	-20.0	-45.0	+ 6.5	-24.4	- 4.2	-15.6
F	+ 9.3	- 2.1	+ 8.6	+ 6.7	+ 5.3	+ 5.3
G	-25.0	- 9.6	+ 5.0	+ 6.6	- 5.7	- 0.8
H	-13.1	+ 2.0	-14.6	+ 0.2	- 7.0	- 1.2
J	+10.0	+29.3	+ 9.0	+13.7	- 1.1	+ 5.9
K	-11.7	+12.6	- 6.7	+ 1.4	- 4.1	- 1.1
L	+22.8	-40.9	+ 9.8	- 3.2	+12.3	- 3.6
M	+16.3	+ 4.2	+23.8	+ 3.4	+ 3.4	+ 9.3
N	+12.8	+21.7	+ 1.2	+12.7	+ 0.1	+ 6.0
O	+ 1.6	+13.3	- 0.9	+11.5	+ 0.4	+ 4.5
P	+21.8	-23.8	- 4.0	- 9.2	+ 4.7	- 8.0

TABLE 5

Experiment #1 - Discharge Data and Responses

Cell No.	Temp.	Discharge Rate at 3.0 v	Max. CCV	Average Volts to 2.0 v	Y <sub>2</sub> Amp-Hrs/Lb CuF <sub>2</sub> to 2.0 v	Y <sub>4</sub> Watt-Hrs/Lb to 2.0 v	Y <sub>6</sub> Watt-Hrs/Cm <sup>3</sup> to 2.0 v
F-13	+30°C	20 ma/cm <sup>2</sup>	2.70	2.54	146.0	101.0	.597
F-15	+10°C	20 ma/cm <sup>2</sup>	2.90	2.65	75.0	26.2	.279
F-16	+10°C	30 ma/cm <sup>2</sup>	1.90	-	-	-	-
F-18	+10°C	20 ma/cm <sup>2</sup>	2.82	2.48	124.0	61.8	.286
F-19	+10°C	30 ma/cm <sup>2</sup>	2.89	2.40	50.2	26.9	.202
F-20	+30°C	20 ma/cm <sup>2</sup>	2.88	2.63	143.0	78.1	.501
F-21	+30°C	30 ma/cm <sup>2</sup>	2.66	2.39	39.4	16.4	.112
F-22	+10°C	20 ma/cm <sup>2</sup>	2.38	2.33	5.30	1.97	.012
F-23	+30°C	30 ma/cm <sup>2</sup>	2.58	2.43	89.6	23.2	.215
F-24	+30°C	20 ma/cm <sup>2</sup>	2.91	2.72	39.4	22.7	.134
F-25	+10°C	30 ma/cm <sup>2</sup>	2.55	2.36	102.0	38.6	.369
F-27	+10°C	30 ma/cm <sup>2</sup>	2.47	2.33	102.0	64.1	.302
F-28	+10°C	20 ma/cm <sup>2</sup>	2.89	2.65	108.0	39.1	.384
F-29	+30°C	20 ma/cm <sup>2</sup>	2.72	2.46	85.5	36.9	.260
F-31	+30°C	30 ma/cm <sup>2</sup>	2.50	2.34	104.0	38.0	.275
F-32	+30°C	20 ma/cm <sup>2</sup>	2.57	2.35	107.0	41.9	.267
F-33	+10°C	30 ma/cm <sup>2</sup>	2.84	2.45	44.4	26.2	.187
F-34	+30°C	20 ma/cm <sup>2</sup>	2.40	2.29	126.0	37.9	.255
F-54	+10°C	30 ma/cm <sup>2</sup>	2.07	2.03	6.6	1.04	.005
F-56	+30°C	30 ma/cm <sup>2</sup>	2.54	2.28	125.0	91.9	.422

TABLE 6

Experiment #2 - Discharge Data and Responses

Cell No.	Temp.	Discharge Rate at 3.0 v	Max. CCV	Average Volts to 2.0 v	Y <sub>2</sub> Amp-Hrs/Lb CuF <sub>2</sub> to 2.0 v	Y <sub>4</sub> Watt-Hrs/Lb to 2.0 v	Y <sub>6</sub> Watt-Hrs/Cm <sup>3</sup> to 2.0 v
F-62	+30°C	20 ma/cm <sup>2</sup>	2.92	2.70	142.0	79.2	.565
F-47	+10°C	30 ma/cm <sup>2</sup>	2.65	2.36	109.0	48.7	.355
F-63	+30°C	20 ma/cm <sup>2</sup>	2.65	2.54	143.0	52.6	.363
F-52	+10°C	30 ma/cm <sup>2</sup>	2.42	2.30	108.0	32.1	.245
F-55	+30°C	20 ma/cm <sup>2</sup>	2.85	2.66	174.0	77.1	.522
F-60	+10°C	30 ma/cm <sup>2</sup>	2.46	2.29	80.0	34.8	.210
F-48	+30°C	20 ma/cm <sup>2</sup>	2.49	2.35	119.0	31.7	.214
F-57	+10°C	30 ma/cm <sup>2</sup>	1.99	-	-	-	-
F-64	+10°C	20 ma/cm <sup>2</sup>	2.56	2.33	123.0	22.2	.322
F-61	+30°C	30 ma/cm <sup>2</sup>	2.90	2.73	159.0	74.9	.499
F-46	+10°C	20 ma/cm <sup>2</sup>	2.43	2.26	31.5	9.54	.108
F-53	+30°C	30 ma/cm <sup>2</sup>	1.92	-	-	-	-
F-44	+10°C	20 ma/cm <sup>2</sup>	2.45	2.32	140.0	47.1	.243
F-50	+30°C	30 ma/cm <sup>2</sup>	2.28	2.09	66.7	10.9	.109
F-58	+10°C	20 ma/cm <sup>2</sup>	2.00	2.00	3.23	1.72	.0081
F-45	+30°C	30 ma/cm <sup>2</sup>	1.78	-	-	-	-
F-65	+10°C	30 ma/cm <sup>2</sup>	2.49	2.34	129.0	42.6	.379
F-51	+10°C	30 ma/cm <sup>2</sup>	1.91	-	-	-	-
F-43	+10°C	20 ma/cm <sup>2</sup>	2.62	2.31	36.2	9.51	.120
F-49	+30°C	30 ma/cm <sup>2</sup>	1.68	-	-	-	-

TABLE 7

Experiment #3 Mean-Effect Levels - Yates Method

<u>y<sub>2</sub> = Amp-Hrs/Lb of CuF<sub>2</sub> to 2.0 v</u>		<u>y<sub>4</sub> = Watt-Hrs/Lb to 2.0 v</u>		<u>y<sub>6</sub> = Watt-Hrs/Cm<sup>3</sup> to 2.0 v</u>	
Factor	Mean-Effect	Factor	Mean-Effect	Factor	Mean-Effect x 100
B	-31.76	L	+42.38	L	+19.15
C	-29.26	B	-26.73	B	-16.63
A	+20.51	C	-19.23	C	-14.95
M	+19.76	AC	+15.13	AC	+ 9.90
AC	+12.62	A	+ 7.38	D	- 8.88
BD	+11.88	BD	+ 5.99	A	+ 6.63
AD	- 9.63	M	+ 5.44	M	+ 4.73
L	- 9.49	P	- 5.26	AD	- 3.73
H	+ 7.13	O	- 3.94	P	- 3.60
O	- 4.51	AD	- 3.64	BD	+ 3.13
E	+ 3.62	N	- 2.76	O	- 3.10
P	- 3.37	D	- 1.78	CD	- 3.00
D	+ 2.99	E	- 1.68	H	- 1.45
CD	+ 2.38	H	- 1.33	E	+ 1.38
N	+ 0.76	CD	+ 0.59	N	+ 0.25

TABLE 8

Experiment #3 - Discharge Data and Responses

Cell No.	Temp.	Discharge Rate at 3.0 v	Max. CCV	Average Volts to 2.0 v	Y <sub>2</sub> Amp-Hrs/Lb CuF <sub>2</sub> to 2.0 v	Y <sub>4</sub> Watt-Hrs/Lb to 2.0 v	Y <sub>6</sub> Watt-Hrs/Cm <sup>3</sup> to 2.0 v
F-82	+20°C	10 ma/cm <sup>2</sup>	3.08	2.77	153.0	66.9	.532
F-95	+40°C	10 ma/cm <sup>2</sup>	3.08	2.79	193.0	132.0	.925
F-85	+20°C	20 ma/cm <sup>2</sup>	3.03	2.54	153.0	92.4	.640
F-79	+40°C	20 ma/cm <sup>2</sup>	3.06	2.71	160.0	57.0	.482
F-80	+20°C	10 ma/cm <sup>2</sup>	2.97	2.70	136.0	103.0	.636
F-90	+40°C	10 ma/cm <sup>2</sup>	2.97	2.76	163.0	50.6	.397
F-87	+20°C	20 ma/cm <sup>2</sup>	2.72	2.40	143.0	44.7	.375
F-98	+40°C	20 ma/cm <sup>2</sup>	2.64	2.47	111.0	78.9	.474
F-93	+20°C	10 ma/cm <sup>2</sup>	3.14	2.91	180.0	72.2	.462
F-89	+40°C	10 ma/cm <sup>2</sup>	3.09	2.76	187.0	124.0	.746
F-96	+20°C	20 ma/cm <sup>2</sup>	2.48	2.30	119.0	72.0	.403
F-91	+40°C	20 ma/cm <sup>2</sup>	3.00	2.88	190.0	71.0	.481
F-81	+20°C	10 ma/cm <sup>2</sup>	2.78	2.61	159.0	110.0	.611
F-92	+40°C	10 ma/cm <sup>2</sup>	3.00	2.72	174.0	58.8	.429
F-84	+20°C	20 ma/cm <sup>2</sup>	2.68	2.29	122.0	37.7	.262
F-97	+40°C	20 ma/cm <sup>2</sup>	2.63	2.46	122.0	67.8	.404
F-94	+40°C	10 ma/cm <sup>2</sup>	3.15	2.91	196.0	137.0	.977
F-86	+20°C	20 ma/cm <sup>2</sup>	2.60	2.41	143.0	42.8	.385
F-88	+40°C	10 ma/cm <sup>2</sup>	3.13	2.89	199.0	138.0	.822
F-83	+20°C	20 ma/cm <sup>2</sup>	2.42	2.26	78.9	23.0	.154

TABLE 9

Experiment #4 Cell Construction Characteristics

Cell Nos.	Test Nos.	Temperature A	Discharge Rate B	Electrolyte Molarity E
F-103 & F-104	1 & 2 *	+31 °C	19	2.0
F-105 & F-106	3 & 4	+33 °C	18	1.9
F-107 & F-108	5 & 6	+35 °C	17	1.8
F-109 & F-110	7 & 8	+37 °C	16	1.7
F-111 & F-112	9 & 10	+39 °C	15	1.6
F-113 & F-114	11 & 12	+41 °C	14	1.5
F-115 & F-116	13 & 14	+43 °C	13	1.4

The remaining factors were constant for all cells:

Electrolyte Volume	110%
Cathode Thickness	.060"
Cathode Conductor Material	Asbury
Cathode Conductor Content	10%
Cathode Binder Content	2%
Cathode H <sub>2</sub> O Content	8%
Screen Geometry	Exmet 5Ag 14-1/0
Separator Material	Glass
Separator Thickness	.015"
Anode Capacity	125% of Cathode
Electrode Size	2-3/4" x 4"
No. of Electrodes	3

\* Two cells are discharged for each combination of characteristics.

TABLE 10

Experiment #4 - Discharge Data and Responses

Cell No.	Temp.	Discharge Rate at 3.0 v	Max. CCV	Average Volts to 2.0 v	Y <sub>2</sub> Amp-Hrs/Lb CuF <sub>2</sub> to 2.0 v	Y <sub>4</sub> Watt-Hrs/Lb to 2.0 v	Y <sub>6</sub> Watt-Hrs/Cm <sup>3</sup> to 2.0 v
F-103	+31°C	19 ma/cm <sup>2</sup>	2.99	2.75	173.0	83.8	.556
F-104	+31°C	19 ma/cm <sup>2</sup>	3.04	2.81	164.0	91.6	.532
F-105	+33°C	18 ma/cm <sup>2</sup>	3.14	2.97	178.0	89.5	.632
F-106	+33°C	18 ma/cm <sup>2</sup>	3.03	2.82	154.0	81.6	.506
F-107	+35°C	17 ma/cm <sup>2</sup>	3.05	2.84	182.0	83.5	.623
F-108	+35°C	17 ma/cm <sup>2</sup>	3.02	2.76	177.0	83.4	.610
F-109	+37°C	16 ma/cm <sup>2</sup>	3.05	2.81	154.0	76.7	.537
F-110	+37°C	16 ma/cm <sup>2</sup>	3.05	2.82	137.0	65.9	.467
F-111	+39°C	15 ma/cm <sup>2</sup>	3.15	2.92	189.0	94.0	.642
F-112	+39°C	15 ma/cm <sup>2</sup>	3.00	2.77	149.0	74.3	.507
F-113	+41°C	14 ma/cm <sup>2</sup>	3.08	2.81	156.0	79.6	.553
F-114	+41°C	14 ma/cm <sup>2</sup>	3.11	2.84	173.0	91.7	.668
F-115	+43°C	13 ma/cm <sup>2</sup>	3.12	2.89	166.0	90.9	.587
F-116	+43°C	13 ma/cm <sup>2</sup>	3.13	2.87	164.0	86.9	.594



TABLE 11

Experiment #6 Cell Construction Characteristics

Cell Nos.	Test No.	Temp.	Discharge Rate ma/cm <sup>2</sup>	Separator Thickness	Electrolyte Molarity	Cathode H <sub>2</sub> O Content	Cathode Conductor Content
F-133, F-134, F-135	1	+25°C	15	.029"	2.7	10%	10%
F-136, F-137, F-138	2	+30°C	13	.027"	2.5	8%	9%
F-150, F-140, F-141	3	+35°C	11	.025"	2.3	6%	8%
F-142, F-143, F-144	4	+40°C	9	.023"	2.1	4%	7%
F-151, F-146, F-147	5	+45°C	7	.021"	1.9	2%	6%

The remaining factors were constant for all cells:

Electrolyte Volume	110% Full
Cathode Thickness	.080"
Cathode Conductor Material	Asbury Ceylon Graphite
Cathode Binder Content	2%
Screen Geometry	Standard Exmet 5Ag 14-1/0
Separator Material	Glass Fiber Filter Mat
	Two layers of material used at cathode/anode interface.
Anode Capacity	150% of Cathode
Electrode Size	2-3/4" x 4"
No. of Electrodes	3

TABLE 12

Experiment #6 - Discharge Data and Responses

Cell No.	Temp.	Discharge Rate at 3.0 v	Max. CCV	Average Volts to 2.0 v	Y <sub>2</sub> Amp-Hrs/Lb CuF <sub>2</sub> to 2.0 v	Y <sub>4</sub> Watt-Hrs/Lb to 2.0 v	Y <sub>6</sub> Watt-Hrs/Cm <sup>3</sup> to 2.0 v
F-133	+25°C	15 ma/cm <sup>2</sup>	2.79	2.62	160.0	78.9	.511
F-134	+25°C	15 ma/cm <sup>2</sup>	2.70	2.50	158.0	73.2	.466
F-135	+25°C	15 ma/cm <sup>2</sup>	2.98	2.77	181.0	92.0	.577
F-136	+30°C	13 ma/cm <sup>2</sup>	2.93	2.69	162.0	93.5	.554
F-137	+30°C	13 ma/cm <sup>2</sup>	2.94	2.71	187.0	102.0	.589
F-138	+30°C	13 ma/cm <sup>2</sup>	3.04	2.81	181.0	105.0	.621
F-150	+35°C	11 ma/cm <sup>2</sup>	3.09	2.77	182.0	105.0	.634
F-140	+35°C	11 ma/cm <sup>2</sup>	3.15	2.94	182.0	114.0	.696
F-141	+35°C	11 ma/cm <sup>2</sup>	3.20	3.02	204.0	133.0	.802
F-142	+40°C	9 ma/cm <sup>2</sup>	3.25	3.02	197.0	134.0	.834
F-143	+40°C	9 ma/cm <sup>2</sup>	3.27	3.11	203.0	139.0	.887
F-144	+40°C	9 ma/cm <sup>2</sup>	3.25	3.00	197.0	134.0	.830
F-151	+45°C	7 ma/cm <sup>2</sup>	3.30	3.02	191.0	144.0	.836
F-146	+45°C	7 ma/cm <sup>2</sup>	3.28	3.04	163.0	115.0	.739
F-147	+45°C	7 ma/cm <sup>2</sup>	3.29	3.04	178.0	126.0	.776

TABLE 13

Factor Levels for Experiments #7, #8, and #9

Experiment #7 - Use of Copper Screens in Cathodes (5Cu 14-1/0)

Cell #F-162, #F-163, and #F-164

Experiment #8 - Multi-Plate Cells

Cell #F-165  
Cell #F-166 - 5 Plates

Cell #F-167  
Cell #F-168 - 7 Plates

Experiment #9 - 2" x 3" Plates

Cell #F-169, #F-170, and #F-171

Temperature	+40 °C
Discharge Rate	9 ma/cm <sup>2</sup>
Electrolyte Molarity	2.1
Electrolyte Volume	110% Full
Cathode Thickness	.080"
Cathode Conductor Material	Asbury
Cathode Conductor Content	7%
Cathode Binder Content	2%
Cathode H <sub>2</sub> O Content	4%
Screen Geometry	5Ag 14-1/0 (Varied in Exp. #7)
Separator Material	Glass
Separator Thickness	.023"
Anode Capacity	150% of Cathode
Electrode Size	2-3/4" x 4" (Varied in Exp. #9)
No. of Electrodes	3 (Varied in Exp. #8)

TABLE 14

Discharge Data and Responses

Cell No.	Temp.	Discharge Rate at 3.0 v	Max. CCV	Average Volts to 2.0 v	$Y_2$ Amp-Hrs/Lb CuF <sub>2</sub> to 2.0 v	$Y_4$ Watt-Hrs/Lb to 2.0 v	$Y_6$ Watt-Hrs/Cm <sup>3</sup> to 2.0 v
<u>Experiment #7 - Use of Copper Screens in Cathodes</u>							
F-162	+40°C	9 ma/cm <sup>2</sup>	3.24	2.94	162.0	115.0	.679
F-163	+40°C	9 ma/cm <sup>2</sup>	3.25	2.91	176.0	121.0	.673
F-164	+40°C	9 ma/cm <sup>2</sup>	3.23	2.90	165.0	118.0	.675
<u>Experiment #8 - Multi-Plate Cells at Optimum Conditions (5 Plates)</u>							
F-165	+40°C	9 ma/cm <sup>2</sup>	3.20	2.89	167.0	130.0	.712
F-166	+40°C	9 ma/cm <sup>2</sup>	3.22	2.94	189.0	151.0	.839
<u>Experiment #8 - Multi-Plate Cells at Optimum Conditions (7 Plates)</u>							
F-167	+40°C	9 ma/cm <sup>2</sup>	3.23	2.99	194.0	181.0	.883
F-168	+40°C	9 ma/cm <sup>2</sup>	3.25	2.98	195.0	180.0	.876
<u>Experiment #9 - 2" x 3" Plate Cells at Optimum Conditions</u>							
F-169	+40°C	9 ma/cm <sup>2</sup>	3.24	2.99	189.0	92.2	.828
F-170	+40°C	9 ma/cm <sup>2</sup>	3.25	2.99	197.0	95.8	.856
F-171	+40°C	9 ma/cm <sup>2</sup>	3.25	3.03	203.0	98.7	.873

TABLE 15

Experiment #13 - Discharge Data and Responses

Cell No.	Temp.	Discharge Rate at 3.0 v	Max. CCV	Average Volts to 2.0 v	<sup>y2</sup> Amp-Hrs/Lb CuF <sub>2</sub> to 2.0 v	<sup>y4</sup> Watt-Hrs/Lb to 2.0 v	<sup>y6</sup> Watt-Hrs/Cm <sup>3</sup> to 2.0 v
F-188	+40°C	10 ma/cm <sup>2</sup>	3.27	3.12	206.6	123.3	.825
F-189	+40°C	10 ma/cm <sup>2</sup>	3.18	3.01	184.5	90.2	.675
F-190	+40°C	20 ma/cm <sup>2</sup>	3.12	2.90	165.5	104.2	.634
F-191	+40°C	20 ma/cm <sup>2</sup>	3.04	2.78	160.5	98.0	.615
F-192	+40°C	30 ma/cm <sup>2</sup>	2.83	2.58	143.5	56.6	.400
F-193	+40°C	30 ma/cm <sup>2</sup>	2.48	2.32	88.2	34.4	.211

TABLE 16

Experiment #11 Cell Construction Characteristics

Cell No.	Test No.	Separator Thickness D	Electrolyte Molarity E	Cathode Thickness L	Cathode H <sub>2</sub> O Content M
F-172	1a	.023"	2.1	.100"	5%
F-173	1b	.023"	2.1	.100"	5%
F-174	1c	.023"	2.1	.100"	5%
F-175	2a	.022"	2.2	.080"	6%
F-176	2b	.022"	2.2	.080"	6%
F-177	2c	.022"	2.2	.080"	6%
F-178	3a	.021"	2.3	.060"	7%
F-179	3b	.021"	2.3	.060"	7%
F-180	3c	.021"	2.3	.060"	7%
F-181	4a	.020"	2.4	.040"	8%
F-182	4b	.020"	2.4	.040"	8%
F-183	4c	.020"	2.4	.040"	8%

The remaining factors were constant for all cells:

A	Temperature	+15°C
B	Discharge Rate	30 ma/cm <sup>2</sup>
F	Electrolyte Volume	110% Full
N	Cathode Conductor Material	Asbury
O	Cathode Conductor Content	8%
P	Cathode Binder Content	2%
H	Screen Geometry	5Ag 14-1/0
C	Separator Material	Glass
K	Anode Capacity	125% of Cathode
G	Electrode Size	2-3/4" x 4"
J	No. of Electrodes	3

TABLE 17

Experiment #11 - Discharge Data and Responses

Cell No.	Temp.	Discharge Rate at 3.0 v	Max. CCV	Average Volts to 2.0 v	Y <sub>2</sub> Amp-Hrs/Lb CuF <sub>2</sub> to 2.0 v	Y <sub>4</sub> Watt-Hrs/Lb to 2.0 v	Y <sub>6</sub> Watt-Hrs/Cm <sup>3</sup> to 2.0 v
F-172	+15°C	30 ma/cm <sup>2</sup>	2.36	2.18	29.6	15.8	.101
F-173	+15°C	30 ma/cm <sup>2</sup>	2.08	2.04	12.2	6.1	.040
F-174	+15°C	30 ma/cm <sup>2</sup>	2.39	2.26	69.1	38.0	.250
Average			2.28	2.16	37.0	20.0	.130
F-175	+15°C	30 ma/cm <sup>2</sup>	2.44	2.24	43.0	20.7	.136
F-176	+15°C	30 ma/cm <sup>2</sup>	2.41	2.27	58.3	28.7	.193
F-177	+15°C	30 ma/cm <sup>2</sup>	2.18	2.10	34.9	16.0	.106
Average			2.34	2.20	45.4	21.8	.145
F-178	+15°C	30 ma/cm <sup>2</sup>	2.68	2.45	113.0	51.7	.341
F-179	+15°C	30 ma/cm <sup>2</sup>	2.55	2.34	96.1	41.6	.276
F-180	+15°C	30 ma/cm <sup>2</sup>	2.38	2.23	60.0	25.0	.161
Average			2.54	2.34	89.7	39.4	.259
F-181	+15°C	30 ma/cm <sup>2</sup>	2.62	2.45	132.0	38.9	.332
F-182	+15°C	30 ma/cm <sup>2</sup>	2.34	2.21	71.9	19.4	.165
F-183	+15°C	30 ma/cm <sup>2</sup>	2.31	2.21	73.2	19.9	.165
Average			2.42	2.29	92.4	26.1	.221

TABLE 19

Experiment #12a Cell Construction Characteristics

Cell Nos.	Separator Thickness A	Electrolyte Molarity B	Cathode Thickness C	Cathode H <sub>2</sub> O Content D	Cathode Conductor Content E
F-194 & F-232	.020"	2.1	.035"	4%	7%
F-233 & F-234	.030"	2.1	.035"	4%	11%
F-235 & F-199	.020"	2.7	.035"	4%	11%
F-201, F-236 & F-237	.030"	2.7	.035"	4%	7%
F-202 & F-203	.020"	2.1	.055"	4%	11%
F-204 & F-205	.030"	2.1	.055"	4%	7%
F-206 & F-207	.020"	2.7	.055"	4%	7%
F-208 & F-209	.030"	2.7	.055"	4%	11%
F-210 & F-239	.020"	2.1	.035"	8%	11%
F-212, F-213 & F-240	.030"	2.1	.035"	8%	7%
F-215 & F-242	.020"	2.7	.035"	8%	7%
F-216 & F-217	.030"	2.7	.035"	8%	11%
F-218 & F-219	.020"	2.1	.055"	8%	7%
F-220 & F-221	.030"	2.1	.055"	8%	11%
F-222 & F-223	.020"	2.7	.055"	8%	11%
F-224 & F-225	.030"	2.7	.055"	8%	7%
F-226 to F-231	.025"	2.4	.045"	6%	9%

The remaining factors were constant for all cells:

Temperature	+10°C
Discharge Rate	38 ma/cm <sup>2</sup> at 3.0 volts
Electrolyte Volume	45 cc
Cathode Conductor Material	Asbury
Cathode Binder Content	2%
Screen Geometry	Distex 5Ag 14-1/0
Separator Material	Glass
Anode Capacity	150% of Cathode
Electrode Size	2" x 3"
No. of Electrodes	3



TABLE 20

Experiment #12a - Discharge Data and Responses

Cell No.	Initial CCV	Max. CCV	Avg. Voltage		Amp-Hrs/Lb CuF <sub>2</sub>		Whrs/Cm <sup>3</sup> Cell	
			2.0 v	1.0 v	y <sub>1</sub> = 2.0 v	y <sub>2</sub> = 1.0 v	y <sub>5</sub> = 2.0 v	y <sub>6</sub> = 1.0 v
F-194	1.78	1.78	-	1.41	-	117	-	.166
F-232	0.04	0.34	-	-	-	-	-	-
F-233	0.08	1.42	-	1.26	-	70.8	-	.073
F-234	0.03	0.59	-	-	-	-	-	-
F-235	0.10	1.14	-	1.10	-	50.9	-	.045
F-199	2.47	2.47	2.19	1.74	73	163	.155	.274
F-201	0.08	0.08	-	-	-	-	-	-
F-236	0.04	0.22	-	-	-	-	-	-
F-237	0.04	0.18	-	-	-	-	-	-
F-202	1.39	2.39	2.27	1.71	47	130	.129	.270
F-203	0.97	2.16	2.12	1.66	32	90	.080	.177
F-204	0.15	1.47	-	1.28	-	58	-	.080
F-205	0.02	0.92	-	-	-	-	-	-
F-206	0.46	1.47	-	1.30	-	104	-	.164
F-207	0.02	1.67	-	1.44	-	130	-	.223
F-208	0.58	1.83	-	1.50	-	120	-	.187
F-209	0.46	1.79	-	1.48	-	160	-	.236
F-210	0.09	2.22	2.12	1.80	103	187	.183	.283
F-239	0.27	2.60	2.41	1.96	120	189	.256	.329
F-212	0.02	0.12	-	-	-	-	-	-
F-213	0.01	0.05	-	-	-	-	-	-
F-240	0.08	0.18	-	-	-	-	-	-
F-215	0.48	1.68	-	1.40	-	164	-	.202
F-242	0.29	2.33	2.22	1.81	102	188	.204	.307
F-216	0.08	1.74	-	1.50	-	148	-	.143

TABLE 20 (con't)

Experiment #12a - Discharge Data and Responses

Cell No.	Initial CCV	Max. CCV	Avg. Voltage		Amp-Hrs/Lb $\text{CuF}_2$		Whrs/ $\text{Cm}^3$ Cell	
			2.0 v	1.0 v	$y_1 = 2.0 \text{ v}$	$y_2 = 1.0 \text{ v}$	$y_5 = 2.0 \text{ v}$	$y_6 = 1.0 \text{ v}$
F-217	0.14	1.77	-	1.55	-	173	-	.191
F-218	0.02	1.74	-	1.45	-	112	-	.179
F-219	0.03	2.33	2.23	1.80	83.5	168	.196	.317
F-220	0.06	2.19	2.12	1.65	38.5	149	.077	.232
F-221	0.09	2.26	2.18	1.72	67.5	168	.132	.258
F-222	0.20	2.20	2.13	1.76	95.3	190	.199	.328
F-223	0.25	2.10	2.06	1.70	63.1	186	.138	.338
F-224	0.02	1.88	-	1.54	-	186	-	.261
F-225	0.62	2.10	2.06	1.60	12.9	182	.026	.281
F-226	0.05	1.26	-	1.16	-	56.4	-	.057
F-227	0.03	1.20	-	1.11	-	46.4	-	.043
F-228	0.04	1.84	-	1.51	-	142	-	.202
F-229	0.06	1.74	-	1.45	-	130	-	.176
F-230	0.03	1.90	-	1.51	-	147	-	.211
F-231	0.04	1.59	-	1.34	-	119	-	.145

TABLE 21

Experiment #14 Cell Construction Characteristics

Cell Nos.	Discharge Rate	Type Carbon	Cathode Conductor Content	Cathode Thickness	Separator Thickness	Electrolyte Molarity	Cathode H <sub>2</sub> O Content	Cathode Binder Content
<u>Experiment #14a</u>								
F-244 & F-245	38 ma/cm <sup>2</sup>	Conductex	11%	.055"	.020"	2.7	8%	2.0%
<u>Experiment #14b</u>								
F-249 & F-250	38 ma/cm <sup>2</sup>	Conductex	14%	.080"	.015"	3.0	10%	1.0%
F-252 & F-253	38 ma/cm <sup>2</sup>	Asbury	14%	.080"	.015"	3.0	10%	1.0%
<u>Experiment #14c</u>								
F-255 * & F-256 *	38 ma/cm <sup>2</sup>	Conductex	11%	.055"	.015"	2.7	8%	0.5%
F-257 ** & F-258	38 ma/cm <sup>2</sup>	Conductex	11%	.055"	.015"	2.7	8%	0.5%
<u>Experiment #14d</u>								
F-259 & F-260	20 ma/cm <sup>2</sup>	Conductex	11%	.055"	.015"	2.7	8%	0.5%
F-261 & F-262	30 ma/cm <sup>2</sup>	Conductex	11%	.055"	.015"	2.7	8%	0.5%
F-263 & F-264	30 ma/cm <sup>2</sup>	Conductex	11%	.055"	.010"	2.7	8%	0.5%
F-265 & F-266	20 ma/cm <sup>2</sup>	Conductex	11%	.055"	.010"	2.7	2%	0.5%
F-267 & F-268	30 ma/cm <sup>2</sup>	Conductex	11%	.055"	.015"	2.7	2%	0.5%
F-269 & F-270	30 ma/cm <sup>2</sup>	Conductex	11%	.055"	.010"	2.7	2%	0.5%
F-271 & F-272	20 ma/cm <sup>2</sup>	Conductex	11%	.055"	.015"	2.7	2%	0.5%
F-273 & F-274	20 ma/cm <sup>2</sup>	Conductex	11%	.055"	.010"	2.7	8%	0.5%

\* Constant "I"

\*\* Constant "R"

TABLE 21 (con't)

Experiment #14 Cell Construction Characteristics

The remaining factors were constant for all cells:

Temperature	+10°C
Electrolyte Volume	45 cc
Screen Geometry	Distex 5Ag 14-1/0
Separator Material	Glass
Anode Capacity	150% of Cathode
Electrode Size	2" x 3"
No. of Electrodes	3

TABLE 22

Experiment #14a, b, and c - Discharge Data and Responses

Cell No.	Initial	Max.	Avg. Voltage		Amp-Hrs/Lb CuF <sub>2</sub>		Whrs/Cm <sup>3</sup> Cell	
	CCV	CCV	2.0 v	1.0 v	y <sub>1</sub> = 2.0 v	y <sub>2</sub> = 1.0 v	y <sub>5</sub> = 2.0 v	y <sub>6</sub> = 1.0 v
<u>Experiment #14a</u>								
F-244	2.24	2.31	2.12	1.67	33.9	214	.078	.386
F-245	2.16	2.16	-	1.65	-	206	-	.358
<u>Experiment #14b</u>								
F-249	2.60	2.60	2.26	1.75	8.0	230	.022	.502
F-250	2.64	2.64	2.33	1.78	5.76	239	.017	.503
F-252	2.24	2.40	2.28	1.42	6.6	181	.017	.297
F-253	2.13	2.36	2.28	1.42	5.46	165	.015	.282
<u>Experiment #14c</u>								
F-255	1.78	2.47	2.12	1.74	59.7	168	.163	.376
F-256	1.87	2.46	2.19	1.93	124	181	.353	.452
F-257	2.27	2.45	2.13	1.76	127	223	.313	.453
F-258	2.43	2.47	2.13	1.86	127	223	.357	.545

TABLE 23

Experiment #14d - Discharge Data and Responses

Cell No.	Initial CCV	Max. CCV	Avg. Voltage		Amp-Hrs/Lb CuF <sub>2</sub>		Whrs/Cm <sup>3</sup> Cell	
			2.0 v	1.0 v	y <sub>1</sub> = 2.0 v	y <sub>2</sub> = 1.0 v	y <sub>5</sub> = 2.0 v	y <sub>6</sub> = 1.0 v
F-259	2.74	2.91	2.79	2.65	110	154	.393	.526
F-260	2.89	2.91	2.79	2.66	135	175	.479	.591
F-261	2.58	2.70	2.58	2.40	35.0	160	.114	.487
F-262	2.33	2.72	2.64	2.44	78.5	177	.254	.529
F-263	2.69	2.70	2.62	2.47	102	176	.354	.575
F-264	2.50	2.71	2.63	2.48	86.4	178	.297	.577
F-265	2.28	2.83	2.74	2.63	134	178	.540	.690
F-266	2.31	2.83	2.75	2.65	141	179	.555	.680
F-267	2.19	2.62	2.57	2.40	77.8	157	.270	.510
F-268	1.98	2.58	2.55	2.41	52.9	131	.176	.410
F-269	2.22	2.65	2.60	2.44	76.6	155	.293	.557
F-270	2.16	2.65	2.60	2.47	94.3	156	.363	.568
F-271	2.30	2.82	2.70	2.57	127	176	.466	.616
F-272	2.31	2.77	2.70	2.61	127	167	.463	.587
F-273	2.82	2.90	2.77	2.67	141	174	.514	.613
F-274	2.85	2.90	2.77	2.71	167	195	.610	.696

TABLE 25

Experiment #15a - Mean-Effect Levels - Yates MethodAmpere-Hours/Lb of  $\text{CuF}_2$ 

<u><math>y_1 - 2.5 \text{ v}</math></u>		<u><math>y_2 - 2.0 \text{ v}</math></u>	
Factor	Mean-Effect	Factor	Mean-Effect
B	-42.3	C	-35.5
C	-36.2	BC	-32.7
A	-27.5	A	-20.0
BD	+10.8	B	-14.8
AB	+ 9.10	AC	-14.5
CD	- 9.05	DE	+12.7
E	+ 7.50	E	+ 9.50
AC	+ 6.70	BD	- 5.50
CE	- 6.35	CE	+ 5.50
BE	+ 5.58	AE	+ 5.50
DE	- 5.18	D	+ 4.75
AD	+ 3.53	BE	+ 4.25
AE	- 3.08	AB	- 4.20
D	- 1.65	CD	- 3.25
BC	- 1.55	AD	+ 2.75

A = Separator Thickness

B = Electrolyte Molarity

C = Cathode Thickness

D = Cathode  $\text{H}_2\text{O}$  Content

E = Cathode Conductor Content

TABLE 26

Experiment #15a Cell Construction Characteristics

Cell Nos.	Separator Thickness A	Electrolyte Molarity B	Cathode Thickness C	Cathode H <sub>2</sub> O Content D	Cathode Conductor Content E
<u>PART A</u>					
F-275 & F-291	.015"	2.3	.060"	8%	8%
F-277, F-301 & F-278	.025"	2.3	.060"	8%	10%
F-279 & F-280	.015"	2.7	.060"	8%	10%
F-281 & F-282	.025"	2.7	.060"	8%	8%
F-283 & F-284	.015"	2.3	.080"	8%	10%
F-285 & F-286	.025"	2.3	.080"	8%	8%
F-303 & F-288	.015"	2.7	.080"	8%	8%
F-289 & F-290	.025"	2.7	.080"	8%	10%
F-292 & F-293	.015"	2.3	.060"	12%	10%
F-294 & F-295	.025"	2.3	.060"	12%	8%
F-296 & F-297	.015"	2.7	.060"	12%	8%
F-298 & F-299	.025"	2.7	.060"	12%	10%
F-300 & F-304	.015"	2.3	.080"	12%	8%
F-305 & F-306	.025"	2.3	.080"	12%	10%
F-307 & F-308	.015"	2.7	.080"	12%	10%
F-309, F-310 & F-315	.025"	2.7	.080"	12%	8%
F-311 & F-312	.020" *	2.5	.070"	10%	9%
F-313 & F-314	.020" **	2.5	.070"	10%	9%

\* One layer material

\*\* Two layers material



TABLE 26 (con't)

Experiment #15a Cell Construction Characteristics

The remaining factors were constant for all cells:

Temperature	+10°C
Discharge Rate	30 ma/cm <sup>2</sup> at 3.0 volts
Cathode Conductor Material	Conductex SC
Cathode Binder Content	.5%
Screen Geometry	Distex 5Ag 14-1/0
Separator Material	Glass
Electrolyte Volume	45 cc
Anode Capacity	150% of Cathode
Electrode Size	2" x 3"
No. of Electrodes	3

TABLE 27

Experiment #15a - Discharge Data and Responses

Cell No.	Initial CCV	Max. CCV	Avg. Voltage		Amp-Hrs/Lb CuF <sub>2</sub>		Whrs/Cm <sup>3</sup> Cell	
			2.5 v	2.0 v	y <sub>1</sub> = 2.5 v	y <sub>2</sub> = 2.0 v	y <sub>5</sub> = 2.5 v	y <sub>6</sub> = 2.0 v
F-275	2.51	2.79	2.68	2.50	106	179	.394	.619
F-291	2.27	2.74	2.67	2.49	91.2	141	.342	.492
F-277	2.57	2.78	2.69	2.38	25.3	90.1	.082	.257
F-301	2.39	2.70	2.62	2.49	103	174	.311	.498
F-278	2.55	2.78	2.61	2.43	67.2	166	.209	.483
F-279	2.64	2.72	2.61	2.45	98.9	176	.340	.566
F-280	2.71	2.72	2.57	2.34	21.1	171	.072	.529
F-281	2.60	2.65	2.54	2.26	8.4	164	.026	.457
F-282	2.61	2.61	2.56	2.29	5.1	168	.016	.460
F-283	2.78	2.78	2.62	2.41	75.2	159	.299	.581
F-284	2.78	2.81	2.71	2.44	77.7	164	.313	.595
F-285	2.33	2.68	2.57	2.37	28.4	103	.096	.323
F-286	2.38	2.65	2.54	2.37	56.2	159	.185	.489
F-303	2.45	2.58	2.55	2.25	6.5	150	.025	.512
F-288	2.05	2.45	-	2.24	-	105	-	.354
F-289	2.28	2.50	-	2.14	-	99.4	-	.285
F-290	2.19	2.43	-	2.10	-	86.9	-	.237
F-292	2.48	2.72	2.62	2.50	111	170	.355	.517
F-293	2.53	2.70	2.60	2.46	94.1	159	.288	.460

TABLE 27 (con't)

Experiment #15a - Discharge Data and Responses

Cell No.	Initial CCV	Max. CCV	Avg. Voltage		Amp-Hrs/Lb CuF <sub>2</sub>		Whrs/Cm <sup>3</sup> Cell	
			2.5 v	2.0 v	y <sub>1</sub> = 2.5 v	y <sub>2</sub> = 2.0 v	y <sub>5</sub> = 2.5 v	y <sub>6</sub> = 2.0 v
F-294	2.20	2.67	2.57	2.35	46.5	140	.138	.379
F-295	2.43	2.60	2.55	2.41	73.7	168	.219	.470
F-296	2.56	2.62	2.57	2.46	88.2	174	.297	.560
F-297	2.20	2.53	2.52	2.36	16.2	167	.053	.513
F-298	2.53	2.67	2.55	2.38	55.3	173	.147	.427
F-299	2.50	2.65	2.54	2.41	34.4	197	.094	.511
F-300	2.62	2.67	2.61	2.41	73.2	163	.281	.575
F-304	2.50	2.66	2.53	2.36	32.3	159	.122	.588
F-305	2.45	2.70	2.61	2.37	20.0	166	.067	.503
F-306	2.64	2.72	2.61	2.33	13.5	148	.045	.432
F-307	2.49	2.61	2.53	2.25	12.8	136	.046	.432
F-308	2.40	2.58	2.54	2.15	6.5	134	.023	.411
F-309	1.99	2.33	-	2.09	-	45.1	-	.124
F-310	2.19	2.44	-	2.21	-	143	-	.406
F-315	2.42	2.48	-	2.36	-	13.5	-	.042
F-311	2.50	2.64	2.56	2.39	59.5	180	.199	.560
F-312	2.51	2.67	2.54	2.36	54.3	175	.184	.548
F-313	2.39	2.54	2.52	2.34	14.4	80.3	.047	.244
F-314	2.41	2.55	2.51	2.34	6.1	94.5	.021	.297

TABLE 28

Experiment #15b Cell Construction Characteristics

Cell Nos.	Separator Thickness A	Electrolyte Molarity B	Cathode Thickness C	Cathode H <sub>2</sub> O Content D	Cathode Conductor Content E
<u>PART B</u>					
F-316 & F-317	.030"	2.5	.070"	10%	9%
F-318 & F-319	.010"	2.5	.070"	10%	9%
F-320 & F-321	.020"	2.9	.070"	10%	9%
F-322 & F-323	.020"	2.1	.070"	10%	9%
F-325 & F-340	.020"	2.5	.090"	10%	9%
F-327 & F-341	.020"	2.5	.050"	10%	9%
F-328 & F-329	.020"	2.5	.070"	14%	9%
F-330 & F-331	.020"	2.5	.070"	6%	9%
F-332 & F-333	.020"	2.5	.070"	10%	11%
F-334 & F-335	.020"	2.5	.070"	10%	7%
F-336 to F-339	.020"	2.5	.070"	10%	9%

The remaining factors were constant for all cells:

Temperature	+10°C
Discharge Rate	30 ma/cm <sup>2</sup> at 3.0 volts
Cathode Conductor Material	Conductex SC
Cathode Binder Content	.5%
Screen Geometry	Distex 5Ag 14-1/0
Separator Material	Glass
Electrolyte Volume	45 cc
Anode Capacity	150% of Cathode
Electrode Size	2" x 3"
No. of Electrodes	3

TABLE 29

Experiment #15b - Discharge Data and Responses

Cell No.	Initial CCV	Max. CCV	Avg. Voltage		Amp-Hrs/Lb CuF <sub>2</sub>		Whrs/Cm <sup>3</sup> Cell	
			2.5 v	2.0 v	y <sub>1</sub> = 2.5 v	y <sub>2</sub> = 2.0 v	y <sub>5</sub> = 2.5 v	y <sub>6</sub> = 2.0 v
F-316	2.52	2.68	2.57	2.35	29.9	152	.090	.421
F-317	2.46	2.67	2.54	2.14	15.5	94.5	.049	.251
F-318	2.81	2.81	2.70	2.39	63.6	160	.269	.600
F-319	2.79	2.80	2.68	2.39	61.7	151	.260	.567
F-320	2.42	2.42	-	2.09	-	117	-	.327
F-321	2.42	2.42	-	2.15	-	110	-	.326
F-322	2.60	2.73	2.59	2.38	52.7	149	.189	.491
F-323	2.76	2.80	2.68	2.46	74.9	129	.275	.431
F-325	2.52	2.58	2.54	2.23	6.2	120	.021	.363
F-340	2.42	2.50	-	2.26	-	128	-	.416
F-327	2.47	2.63	2.56	2.34	45.3	157	.132	.417
F-341	2.62	2.62	2.56	2.41	62.5	160	.183	.438
F-328	2.54	2.67	2.59	2.48	114	188	.373	.589
F-329	2.34	2.67	2.57	2.44	49.9	185	.161	.568
F-330	2.11	2.50	-	2.27	-	91.4	-	.295
F-331	2.32	2.58	2.54	2.30	15.8	100	.057	.329
F-332	2.67	2.67	2.61	2.39	72.0	158	.246	.494
F-333	2.58	2.63	2.59	2.26	13.0	162	.043	.470
F-334	2.07	2.07	-	2.04	-	2.3	-	.007
F-335	2.18	2.18	-	2.13	-	5.0	-	.015
F-336	2.54	2.56	2.52	2.38	70.1	182	.247	.605
F-337	2.47	2.56	2.54	2.38	46.5	180	.161	.583
F-338	2.46	2.50	-	2.33	-	183	-	.582
F-339	2.39	2.44	-	2.31	-	186	-	.586

TABLE 30

Experiments #15c, d, and e Cell Construction Characteristics

Experiment No.	Cell Nos.	Test No.	Separator Thickness	Cathode H <sub>2</sub> O Content
15c	F-357 to F-359	-	.020"	16%
15d	F-375 to F-377	1	.020"	15%
	F-378 to F-380	2	.020"	17%
	F-381 to F-383	3	.020"	19%
15e	F-384 to F-386	1	.010"	16%
	F-387 to F-389	2	.015"	16%
	F-390 to F-392	3	.025"	16%

The remaining factors were constant for all cells:

Temperature	+10°C
Discharge Rate	30 ma/cm <sup>2</sup> at 3.0 volts
Electrolyte Molarity	2.5
Electrolyte Volume	45 cc
Cathode Thickness	.070"
Cathode Conductor Material	Conductex SC
Cathode Conductor Content	9%
Cathode Binder Content	.5%
Screen Geometry	Distex 5Ag 14-1/0
Layers of Separator	1
Separator Material	Glass
Anode Capacity	150% of Cathode
Electrode Size	2" x 3"
No. of Electrodes	3 (1 positive and 2 negative)

TABLE 31

Experiment #15c, d, and e - Discharge Data and Responses

Cell No.	Initial	Max.	Avg. Voltage		Amp-Hrs/Lb CuF <sub>2</sub>		Whrs/Cm <sup>3</sup> Cell	
	CCV	CCV	2.5 v	2.0 v	y <sub>1</sub> = 2.5 v	y <sub>2</sub> = 2.0 v	y <sub>5</sub> = 2.5 v	y <sub>6</sub> = 2.0 v
<u>Experiment #15c</u>								
F-357	2.55	2.62	2.53	2.44	96.8	190	.297	.564
F-358	2.52	2.68	2.61	2.55	151	195	.478	.602
F-359	2.57	2.66	2.59	2.40	23.0	191	.072	.560
<u>Experiment #15d</u>								
F-375	2.44	2.61	2.55	2.40	6.3	177	.020	.527
F-376	2.49	2.56	2.53	2.42	4.7	177	.015	.533
F-377	2.41	2.55	2.53	2.36	6.3	163	.020	.476
F-378	2.32	2.52	2.51	2.40	13.2	180	.038	.502
F-379	2.44	2.58	2.55	2.46	78.5	188	.238	.552
F-380	2.48	2.58	2.53	2.40	25.7	166	.078	.482
F-381	2.56	2.60	2.55	2.42	6.6	187	.019	.525
F-382	2.59	2.60	2.55	2.41	9.1	172	.028	.500
F-383	2.54	2.58	2.53	2.39	6.6	170	.019	.476
<u>Experiment #15e</u>								
F-384	2.62	2.72	2.62	2.59	129	159	.469	.570
F-385	2.66	2.81	2.67	2.58	126	158	.472	.572
F-386	2.58	2.68	2.59	2.56	127	164	.459	.585
F-387	2.55	2.67	2.58	2.51	112	175	.380	.578
F-388	2.63	2.67	2.57	2.50	118	178	.389	.571
F-389	2.61	2.67	2.59	2.52	116	174	.385	.559
F-390	2.48	2.48	-	2.14	-	143	-	.349
F-391	2.60	2.62	2.56	2.32	6.4	173	.019	.459
F-392	2.52	2.56	2.53	2.34	3.9	164	.011	.444

TABLE 32

Experiment #17 Cell Construction Characteristics

Cell Nos.	Test No.	Separator Thickness A	Electrolyte Molarity B	Cathode Thickness C	Cathode Conductor Content D	Cathode H <sub>2</sub> O Content E
F-363 to F-365	1	.021"	2.5	.060"	9.5%	8.8%
F-366 to F-368	2	.019"	2.4	.070"	10.0%	9.0%
F-369 to F-371	3	.017"	2.3	.080"	10.5%	9.2%
F-360 to F-362	4	.015"	2.2	.090"	11.0%	9.4%
F-372 to F-374	5	.013"	2.1	.100"	11.5%	9.6%

The remaining factors were constant for all cells:

Temperature	+10°C
Discharge Rate	30 ma/cm <sup>2</sup> at 3.0 volts
Electrolyte Volume	45 cc
Cathode Conductor Material	Conductex SC
Cathode Conductor Content	.5%
Screen Geometry	Distex 5Ag 14-1/0
Layers of Separator	1
Separator Material	Glass
Anode Capacity	150% of Cathode
Electrode Size	2" x 3"
No. of Electrodes	3 (1 positive and 2 negative)



TABLE 33

Experiment #17 - Discharge Data and Responses

Cell No.	Initial CCV	Max. CCV	Avg. Voltage		Amp-Hrs/Lb CuF <sub>2</sub>		Whrs/Cm <sup>3</sup> Cell	
			2.5 v	2.0 v	y <sub>1</sub> = 2.5 v	y <sub>2</sub> = 2.0 v	y <sub>5</sub> = 2.5 v	y <sub>6</sub> = 2.0 v
F-363	2.66	2.74	2.59	2.46	102	182	.322	.546
F-364	2.18	2.68	2.56	2.43	68.4	174	.214	.518
F-365	2.47	2.70	2.52	2.49	107	177	.345	.542
F-366	2.60	2.68	2.61	2.34	19.1	142	.067	.447
F-367	2.67	2.72	2.56	2.42	64.2	169	.221	.549
F-368	2.48	2.69	2.56	2.32	18.6	163	.063	.503
F-369	2.72	2.72	2.61	2.36	41.6	118	.157	.403
F-370	2.63	2.74	2.59	2.27	15.4	127	.057	.414
F-371	2.55	2.68	2.63	2.36	52.3	116	.197	.393
F-360	2.61	2.74	2.63	2.31	37.6	153	.155	.552
F-361	2.53	2.78	2.62	2.41	74.3	166	.288	.591
F-362	2.58	2.72	2.59	2.21	13.8	139	.055	.474
F-372	2.53	2.76	2.66	2.36	61.5	154	.248	.554
F-373	2.45	2.73	2.61	2.26	34.5	110	.136	.373
F-374	2.50	2.74	2.61	2.34	37.6	136	.151	.488

TABLE 34

Experiment #24 Cell Construction Characteristics

Cell Nos.	Separator Thickness/ Layers	Electrolyte Molarity	Cathode Thickness	Cathode H <sub>2</sub> O Content	Cathode Conductor Content
F-508 to F-510	.020"/1 Layer	2.5	.070"	13%	9%
F-511 to F-513	.025"/1 Layer	2.6	.060"	16%	10%
F-514 to F-516	.030"/1 Layer	2.7	.050"	19%	11%
F-517 to F-519	.030"/2 Layers	2.7	.040"	19%	11%

The remaining factors were constant for all cells:

Temperature	+10°C
Discharge Rate	30 ma/cm <sup>2</sup> at 3.0 volts
Electrolyte Volume	45 cc
Cathode Conductor Material	Conductex
Cathode Binder Content	.5%
Screen Geometry	Distex 5Ag 14-1/0
Separator Material	Glass
Anode Capacity	150% of Cathode
Electrode Size	2" x 3"
No. of Electrodes	3 (1 positive and 2 negative)

TABLE 35

Experiment #24 - Discharge Data and Responses

Cell No.	OCV	Initial CCV	Max. CCV	Avg. Voltage		Discharge Life - Hours		Amp-Hrs/Lb CuF <sub>2</sub>		Whrs/Lb CuF <sub>2</sub>		Whrs/Cm <sup>3</sup> Cell	
				2.5 v	2.0 v	2.5 v	2.0 v	2.5 v	2.0 v	2.5 v	2.0 v	2.5 v	2.0 v
F-508	3.38	2.43	2.63	2.57	2.46	0.70	1.80	68.5	168	176	414	.220	.516
F-509	3.40	2.42	2.61	2.56	2.46	1.00	1.93	96.8	180	248	444	.311	.557
F-510	3.39	2.44	2.68	2.60	2.53	1.37	1.85	137.0	181	356	458	.451	.581
F-511	3.36	2.41	2.58	2.56	2.57	0.12	1.43	14.5	166	37.1	393	.038	.402
F-512	3.35	2.84	2.84	2.56	2.43	0.63	1.48	76.1	169	195.0	411	.203	.428
F-513 *	3.39	2.57	2.57	2.54	2.24	0.08	1.05	9.9	110	25.1	247	.027	.263
F-514 *	3.41	2.39	2.47	-	2.24	-	0.88	-	110	-	247	-	.263
F-515 *	3.41	2.33	2.56	2.54	2.14	0.05	0.92	7.2	111	18.0	238	.016	.205
F-516	3.41	2.43	2.64	2.55	2.47	0.83	1.32	12.2	185	311.0	457	.273	.401
F-517	3.41	2.38	2.55	2.55	2.20	0.05	1.35	7.1	166	18.3	366	.016	.326
F-518	3.41	2.54	2.66	2.60	2.37	0.15	1.55	21.7	204	56.6	482	.050	.429
F-519	3.41	2.45	2.62	2.52	2.44	0.32	1.32	45.2	182	114.0	443	.098	.382

\* Cell temperature profile indicated greater than normal heating suggesting a separator shorting problem.  
Post-test inspection identified significant amounts of copper in the separator material.

TABLE 36

Experiment #16 Cell Construction Characteristics

Experiment No.	Cell Nos.	Separator Thickness/ Layers	Cathode Conductor Material	Cathode Binder Content
16a	F-345 to F-347	.020"/1 Layer	Asbury	2.0%
16b	F-348 to F-350	.020"/1 Layer	Conductex	2.0%
16c	F-351 to F-353	.020"/1 Layer	Asbury	0.5%
16d	F-354 to F-356	.020"/1 Layer	Conductex	0.5%
16e	F-393 to F-395	.023"/2 Layers	Conductex	0.5%

The remaining factors were constant for all cells:

Temperature	+40°C
Discharge Rate	30 ma/cm <sup>2</sup> at 3.0 volts
Electrolyte Molarity	2.1
Electrolyte Volume	45 cc
Cathode Thickness	.080"
Cathode Conductor Content	7%
Cathode H <sub>2</sub> O Content	4%
Screen Geometry	Exmet 5Ag 14-1/0
Separator Material	Glass
Anode Capacity	150% of Cathode
Electrode Size	2" x 3"
No. of Electrodes	3

TABLE 37

Experiment #16 - Discharge Data and Responses

Cell No.	Initial	Max.	Avg. Voltage		Amp-Hrs/Lb CuF <sub>2</sub>		Whrs/Cm <sup>3</sup> Cell	
	CCV	CCV	2.5 v	2.0 v	y <sub>1</sub> = 2.5 v	y <sub>2</sub> = 2.0 v	y <sub>5</sub> = 2.5 v	y <sub>6</sub> = 2.0 v
<u>Experiment #16a</u>								
F-345	2.69	2.83	2.73	2.57	74.6	110	.295	.408
F-346	2.64	2.82	2.71	2.53	51.6	80.1	.219	.317
F-347	2.66	2.81	2.71	2.50	45.8	77.0	.192	.298
<u>Experiment #16b</u>								
F-348	2.78	2.82	2.71	2.54	49.6	74.5	.211	.297
F-349	2.81	2.84	2.71	2.52	27.5	45.3	.119	.183
F-350	2.73	2.73	2.65	2.44	30.2	61.8	.124	.233
<u>Experiment #16c</u>								
F-351	2.60	2.86	2.75	2.61	104	141	.441	.565
F-352	2.66	2.95	2.81	2.64	86.2	112	.375	.456
F-353	2.59	2.83	2.74	2.57	57.6	84.2	.251	.345
<u>Experiment #16d</u>								
F-354	2.68	2.79	2.69	2.54	94.8	141	.403	.566
F-355	2.72	2.77	2.70	2.54	75.0	108	.321	.435
F-356	2.79	2.84	2.75	2.54	93.0	144	.398	.572
<u>Experiment #16e</u>								
F-393	2.77	2.81	2.74	2.58	96.0	132	.385	.501
F-394	Environment Chamber Malfunction After Cell Activated							
F-395	2.92	2.94	2.84	2.74	105	128	.454	.531

TABLE 38

Experiment #19 Cell Construction Characteristics

Experiment No.	Cell Nos.	Test No.	Temperature	Electrolyte Molarity	Cathode Conductor Content	Cathode H <sub>2</sub> O Content
19a	F-429 to F-431	1a, b, c	+10°C	2.7	11%	8%
	F-432 to F-434	2a, b, c	+10°C	2.5	9%	6%
	F-435 to F-437	3a, b, c	+10°C	2.3	7%	4%
19b	F-441 to F-443	1a, b, c	0°C	2.7	11%	8%
	F-444 to F-446	2a, b, c	+5°C	2.7	11%	8%
	F-447 to F-449	3a, b, c	+40°C	2.7	11%	8%
19c	F-450 to F-452	1a, b, c	+10°C	2.3	11%	8%
	F-453 to F-455	2a, b, c	+10°C	2.5	11%	8%

The remaining factors were constant for all cells:

Discharge Rate	20 ma/cm <sup>2</sup> at 2.5 volts
Electrolyte Volume	50 cc
Cathode Thickness	.070"
Cathode Conductor Material	Conductex SC
Cathode Binder Content	.5%
Screen Geometry	Distex 5Ag 14-1/0
Separator Material	Glass, 1 Layer
Separator Thickness	.010"
Anode Capacity	150% of Cathode
Electrode Size	2-3/4" x 4"
No. of Electrodes	3

TABLE 39

Experiment #19a, b, and c - Discharge Data and Responses

Cell No.	Initial	Max.	Avg. Voltage		Amp-Hrs/Lb CuF <sub>2</sub>		Whrs/Cm <sup>3</sup> Cell	
	CCV	CCV	2.5 v	2.0 v	y <sub>1</sub> = 2.5 v	y <sub>2</sub> = 2.0 v	y <sub>5</sub> = 2.5 v	y <sub>6</sub> = 2.0 v
<u>Experiment #19a</u>								
F-429	2.57	2.81	2.68	2.55	114	191	.431	.686
F-430	2.75	2.91	2.75	2.63	126	183	.501	.699
F-431	2.64	2.74	2.66	2.53	114	174	.418	.607
F-432	2.63	2.72	2.62	2.48	86.6	142	.347	.541
F-433	2.69	2.89	2.77	2.55	94.2	155	.409	.618
F-434	2.44	2.82	2.70	2.52	96.2	163	.396	.626
F-435	2.54	2.89	2.75	2.58	82.9	126	.379	.542
F-436	2.52	2.76	2.66	2.48	70.1	119	.303	.480
F-437	2.51	2.64	2.58	2.47	85.0	119	.356	.478
<u>Experiment #19b</u>								
F-441	2.58	2.74	2.65	2.49	100	173	.394	.641
F-442	2.57	2.68	2.62	2.44	74.9	165	.302	.618
F-443	2.65	2.78	2.67	2.54	107	171	.422	.641
F-444	2.53	2.70	2.64	2.54	116	186	.463	.714
F-445	2.58	2.64	2.58	2.47	103	179	.386	.642
F-446	2.60	2.77	2.68	2.55	117	176	.443	.632
F-447	2.99	3.01	2.88	2.80	166	192	.693	.780
F-448	2.99	3.01	2.87	2.79	159	188	.685	.788
F-449	2.99	3.01	2.86	2.79	163	192	.666	.766
<u>Experiment #19c</u>								
F-450	2.76	2.81	2.70	2.62	146	188	.592	.737
F-451	2.80	2.88	2.73	2.65	144	183	.568	.702
F-452	2.83	2.90	2.74	2.61	130	174	.531	.674
F-453	2.80	2.93	2.75	2.62	128	170	.523	.694
F-454	2.81	2.96	2.78	2.72	149	179	.590	.692
F-455	2.76	2.94	2.77	2.67	137	176	.561	.694

TABLE 40

Anhydrous Cell Construction Variables

	Research Group	Experiment #18b	
		F-398	F-427
Plate Area	6 cm <sup>2</sup>	77.4 cm <sup>2</sup>	38.7 cm <sup>2</sup>
No. of Electrodes	2	3	2
Separator - Glass	2 Layers	1 Layer	2 Layers
Cathode Conductor Material	Conductex	Asbury	Conductex
Cathode Conductor Content	9%	7%	9%
Cathode Thickness	.050"	.080"	.050"

The remaining factors were constant for all cells:

Temperature	+40°C
Discharge Rate	10 ma/cm <sup>2</sup> at 3.0 volts
Electrolyte Molarity	2.1
Electrolyte Volume	45 cc for NASA Cells
Cathode Binder Content	.5%
Cathode H <sub>2</sub> O Content	0%
Separator Thickness	.020"

Discharge Results

	Experiment #18b	
	F-398	F-427
OCV	3.40	3.42
ICV	0.23	3.08
MCV	0.23	3.18
Amp-Hrs/Lb CuF <sub>2</sub> to 2.0 v	-	197
Whrs/Lb CuF <sub>2</sub> to 2.0 v	-	595
Whrs/Cm <sup>3</sup> of Cell to 2.0 v	-	0.993



TABLE 41

Experiment #20 Cell Construction Characteristics

Experiment No.	Cell Nos.	Electrolyte Molarity	Cathode Conductor Content
20a	F-438 to F-440	2.1	9%
20b	F-456 to F-458	2.1	12%
	F-459 to F-461	2.1	15%
	F-472 to F-474	2.1	18%
	F-475 to F-477	2.1	21%
20c	F-462 to F-464	2.7	9%
	F-465 to F-467	2.4	9%

The remaining factors were constant for all cells:

Temperature	+40°C
Discharge Rate	10 ma/cm <sup>2</sup> at 3.0 volts
Electrolyte Volume	45 cc
Cathode Thickness	.050"
Cathode Conductor Material	Conductex
Cathode Binder Content	.5%
Cathode H <sub>2</sub> O Content	0%
Screen Geometry	Exmet 5Ag 14-1/0
Separator Material	Glass, 2 Layers
Separator Thickness	.020"
Anode Capacity	150% of Cathode
Electrode Size	2" x 3"
No. of Electrodes	3

TABLE 42

Experiment #20a, b, and c - Discharge Data and Responses

Cell No.	Initial	Max.	Avg. Voltage		Amp-Hrs/Lb CuF <sub>2</sub>		Whrs/Cm <sup>3</sup> Cell	
	CCV	CCV	2.5 v	2.0 v	y <sub>1</sub> = 2.5 v	y <sub>2</sub> = 2.0 v	y <sub>5</sub> = 2.5 v	y <sub>6</sub> = 2.0 v
<u>Experiment #20a</u>								
F-438	3.15	3.18	3.06	2.96	140	159	.571	.627
F-439	3.09	3.18	3.07	3.01	146	162	.604	.661
F-440	3.12	3.16	3.04	2.95	146	162	.600	.644
<u>Experiment #20b</u>								
F-456	3.12	3.16	3.03	2.98	165	177	.632	.664
F-457	3.10	3.23	3.06	2.97	151	164	.586	.617
F-458	3.19	3.24	3.10	3.03	140	155	.550	.593
F-459	3.21	3.23	3.07	3.01	169	180	.634	.662
F-460	3.16	3.23	3.07	3.01	180	189	.663	.686
F-461	3.20	3.24	3.10	3.04	187	195	.684	.701
F-472	3.15	3.23	3.09	3.03	188	198	.667	.688
F-473	3.18	3.21	3.08	2.99	167	181	.621	.652
F-474	3.12	3.16	3.03	2.98	173	186	.617	.652
F-475	3.21	3.27	3.09	3.06	186	195	.673	.698
F-476	3.23	3.25	3.09	3.06	188	199	.692	.726
F-477	3.22	3.25	3.08	3.05	181	191	.650	.680
<u>Experiment #20c</u>								
F-462	3.12	3.19	3.07	3.02	177	189	.690	.724
F-463	3.10	3.19	3.08	3.03	171	184	.697	.735
F-464	3.10	3.19	3.05	2.99	173	188	.682	.729
F-465	3.04	3.14	3.03	2.95	174	180	.683	.688
F-466	3.00	3.15	3.06	3.00	180	192	.684	.720
F-467	3.00	3.13	3.00	2.95	174	190	.683	.735

TABLE 43

Experiment #21 Cell Construction Characteristics

<u>Experiment No.</u>	<u>Cell Nos.</u>	<u>Separator Material</u>	<u>SO<sub>2</sub> Atmosphere</u>
21a	F-478 to F-480	2 Layers Glass + 1 Layer 2974	No
21b	F-481 to F-483	2 Layers Glass	No
21c	F-484 to F-486	2 Layers Glass	Yes
21d	F-487 to F-489	2 Layers Glass + 1 Layer 2974	Yes

The remaining factors were constant for all cells:

Temperature	+40°C
Discharge Rate	10 ma/cm <sup>2</sup> at 3.0 volts
Electrolyte Molarity	2.5
Electrolyte Volume	45 cc
Cathode Thickness	.050"
Cathode Conductor Material	Conductex
Cathode Conductor Content	21%
Cathode Binder Content	.5%
Cathode H <sub>2</sub> O Content	0%
Screen Geometry	Exmet 5Ag 14-1/0
Anode Capacity	150% of Cathode
Electrode Size	2" x 3"
No. of Electrodes	3

TABLE 44

Experiment #21 - Discharge Data and Responses

Cell No.	Initial	Max.	Avg. Voltage		Amp-Hrs/Lb		Whrs/Lb CuF <sub>2</sub>		Whrs/Cm <sup>3</sup> Cell	
	CCV	CCV	2.5 v	2.0 v	2.5 v	2.0 v	2.5 v	2.0 v	2.5 v	2.0 v
<u>Experiment #21a</u>										
F-478	3.18	3.20	2.91	2.77	108	130	316	359	.377	.428
F-479	3.14	3.16	2.88	2.73	88.4	115	254	313	.303	.373
F-480	3.15	3.16	2.84	2.68	89.5	123	254	329	.304	.395
<u>Experiment #21b</u>										
F-481	3.19	3.23	3.10	3.07	187	200	578	613	.623	.662
F-482	3.19	3.23	3.08	3.05	191	201	588	612	.650	.677
F-483	3.20	3.23	3.10	3.07	197	206	612	633	.701	.726
<u>Experiment #21c</u>										
F-484	2.74	3.12	2.99	2.96	202	207	604	614	.680	.691
F-485	2.75	3.16	3.00	2.97	198	203	594	603	.689	.699
F-486 *	2.72	2.86	2.79	2.76	130	139	362	383	.381	.404
<u>Experiment #21d</u>										
F-487	3.05	3.05	2.87	2.58	61.9	98.5	178	254	.205	.293
F-488	3.09	3.09	2.75	2.64	74.4	95.0	204	250	.231	.282
F-489	0.90	0.92	-	-	-	-	-	-	-	-

\* Post-test analyses identified large unactivated areas within cell, the cause of which has been tentatively associated with pockets of gas formed from the flash evaporation of the SO<sub>2</sub> during activation.

TABLE 45

Experiment #22 and #23 Cell Construction Characteristics

Experiment No.	Cell Nos.	Electrode Size	Test Vehicle	Electrolyte Molarity	Type/Mode	SO <sub>2</sub> Atmosphere
22a	F-490 to F-492	2" x 3"	Std. NASA Chamber	2.5	LiBF <sub>4</sub> /MF	Yes
22b	F-493 to F-495	2" x 3"	Std. NASA Chamber	2.1	LiBF <sub>4</sub> /MF	No
	F-496 to F-498	2" x 3"	Std. NASA Chamber	2.5	LiBF <sub>4</sub> /MF	No
	F-499 to F-501	2" x 3"	Std. NASA Chamber	2.7	LiBF <sub>4</sub> /MF	No
23	F-502 to F-504	1½" x 5/8"	Mod. Compatibility Tube	2.5	LiAsF <sub>6</sub> /MF *	No
	F-505 to F-507	1½" x 5/8"	Mod. Compatibility Tube	2.5	LiAsF <sub>6</sub> /MF **	No

The remaining factors were constant for all cells:

Temperature	+40°C
Discharge Rate	10 ma/cm <sup>2</sup> at 3.0 volts
Electrolyte Volume	2" x 3" 45 cc
	1½" x 5/8" (wick) 10 cc
	1½" x 5/8" (flooded) 30 cc
Cathode Thickness	.050"
Cathode Conductor Material	Conductex
Cathode Conductor Content	21%
Cathode Binder Content	.5%
Cathode H <sub>2</sub> O Content	0%
Screen Geometry	Exmet 5Ag 14-1/0
Separator Material	Glass, 2 Layers
Anode Capacity	150% of Cathode
No. of Electrodes	3

\* Wick

\*\* Flooded

TABLE 46

Experiments #22 and #23 - Discharge Data and Responses

Cell No.	OCV	Initial CCV	Max. CCV	Avg. Voltage		Discharge Life - Hours		Amp-Hrs/Lb CuF <sub>2</sub>		Whrs/Lb CuF <sub>2</sub>		Whrs/Cm <sup>3</sup> Cell	
				2.5 v	2.0 v	2.5 v	2.0 v	2.5 v	2.0 v	2.5 v	2.0 v	2.5 v	2.0 v
Experiment #22a													
F-490	3.43	2.78	3.09	2.95	2.93	3.65	3.70	190	191	560	561	.648	.648
F-491	3.44	2.94	3.05	2.91	2.89	3.43	3.48	187	188	544	545	.607	.608
F-492	-	Equipment Malfunction - Environment Chamber Temperature Incorrect											-
Experiment #22b													
F-493	3.44	2.72	3.01	2.87	2.75	2.58	3.28	146	178	419	491	.429	.502
F-494	3.44	2.68	3.01	2.87	2.75	2.40	3.10	124	154	356	422	.404	.480
F-495	3.44	2.61	2.95	2.80	2.67	2.45	3.27	127	162	356	434	.396	.483
F-496	3.45	2.68	2.99	2.85	2.73	2.80	3.65	145	181	412	493	.469	.561
F-497	3.45	2.68	3.03	2.87	2.77	2.93	3.63	148	177	426	491	.500	.576
F-498	3.44	2.60	2.99	2.82	2.72	2.70	3.48	141	175	397	476	.458	.549
F-499	3.44	2.81	3.09	2.92	2.81	2.92	3.65	153	184	445	515	.525	.608
F-500	3.43	2.74	3.02	2.86	2.75	2.70	3.37	145	173	414	476	.458	.526
F-501	3.43	2.67	3.02	2.87	2.75	2.57	3.20	141	168	404	463	.448	.512
Experiment #23													
G-502	3.36	2.97	3.26	3.14	3.11	3.27	3.43	182	190	574	590	.556	.572
G-503	3.42	2.80	3.18	3.07	3.04	3.28	3.45	189	197	581	600	.568	.586
G-504	3.35	2.85	3.23	3.12	3.09	3.28	3.48	181	190	565	588	.558	.581
G-505	3.37	2.84	3.22	3.10	3.06	3.22	3.43	173	183	535	559	.550	.574
G-506 *	3.28	2.68	2.68	2.63	2.39	0.73	1.82	33.4	75.2	87.7	180	.088	.180
G-507	3.42	2.88	3.23	3.12	3.08	3.02	3.27	162	173	505	534	.478	.505

\* Possible intermittent short between electrodes identified during post-test analysis.

TABLE 47

Experiment #26 Cell Construction Characteristics

Experiment No.	Cell Nos.	Temperature	SO <sub>2</sub> Quantity	Discharge Rate
26a	F-520 to F-522	+40°C	0	20 ma/cm <sup>2</sup>
	F-546 to F-548	+40°C	0	25 ma/cm <sup>2</sup>
	F-523 to F-525	+40°C	0	30 ma/cm <sup>2</sup>
26b	F-543 to F-545	+35°C	0	10 ma/cm <sup>2</sup>
26c	F-540 to F-542	+30°C	0	10 ma/cm <sup>2</sup>
	F-617 to F-619	+30°C	.090 g/g elect.	10 ma/cm <sup>2</sup>

The remaining factors were constant for all cells:

Electrolyte Molarity	2.5 M LiAsF <sub>6</sub> /MF
Electrolyte Volume	45 cc
Cathode Thickness	.050"
Cathode Conductor Material	Conductex SC
Cathode Conductor Content	21%
Cathode Binder Content	.5%
Cathode H <sub>2</sub> O Content	0%
Screen Geometry	Exmet 5Ag 14-1/0
	F-617 to F-619 Anode Screen - Exmet 5 Cu 7-1/0
Separator Material	Glass, 2 Layers
Separator Thickness	.020"
Anode Capacity	150% of Cathode
Electrode Size	2" x 3"
No. of Electrodes	3 (1 positive and 2 negative)

TABLE 48

Experiment #26 - Discharge Data and Responses

Cell No.	OCV	Initial	Max.	Avg. Voltage		Discharge		Amp-Hrs/Lb		Whrs/Lb CuF <sub>2</sub>		Whrs/Cm <sup>3</sup>	
		CCV	CCV	2.5 v	2.0 v	Life - Hours		CuF <sub>2</sub>		2.5 v	2.0 v	Cell	
						2.5 v	2.0 v	2.5 v	2.0 v	2.5 v	2.0 v	2.5 v	2.0 v
<u>Experiment #26a</u>													
F-520	3.42	2.57	2.93	2.78	2.74	1.38	1.55	145	160	404	439	.435	.472
F-521	3.42	2.93	3.04	2.98	2.92	1.63	1.77	185	197	553	575	.600	.624
F-522	3.41	2.60	3.08	2.94	2.91	1.68	1.77	196	203	576	592	.607	.625
F-546	3.40	2.43	2.89	2.78	2.73	0.93	1.08	132	151	367	413	.361	.404
F-547	3.35	2.73	2.88	2.80	2.73	0.92	1.07	155	176	435	482	.380	.421
F-548	3.42	2.51	2.70	2.62	2.56	0.98	1.27	115	144	300	369	.345	.425
F-523	3.41	2.47	2.47	-	2.27	-	0.42	-	56.4	-	128	-	.138
F-524	3.41	2.45	2.54	2.53	2.44	0.35	0.92	51.7	131	131	319	.127	.309
F-525	3.41	2.42	2.42	-	2.26	-	0.40	-	57.0	-	129	-	.128
<u>Experiment #26b</u>													
F-543	3.40	3.12	3.14	3.03	2.96	2.77	3.02	183	194	553	575	.513	.533
F-544	3.40	3.10	3.15	3.04	2.97	3.10	3.35	180	191	548	566	.553	.572
F-545	3.40	3.12	3.15	3.03	3.00	2.70	2.92	181	193	549	579	.490	.517
<u>Experiment #26c</u>													
F-540	3.40	2.64	2.66	2.61	2.41	0.52	1.18	26.2	55.6	68.3	134	.067	.132
F-541	3.40	2.73	2.73	2.63	2.44	0.65	1.33	34.0	65.0	89.2	157	.088	.155
F-542	3.40	2.63	2.66	2.61	2.39	0.45	1.07	20.8	45.3	54.4	108	.062	.124
F-617	3.40	3.11	3.17	2.98	2.95	4.17	4.40	166	173	494	513	.625	.649
F-618	3.42	3.01	3.14	2.97	2.96	4.32	4.45	169	173	502	512	.650	.663
F-619	3.42	3.10	3.15	2.99	2.95	4.15	4.48	170	181	508	536	.652	.688



TABLE 49

Experiment #18a and c Cell Construction Characteristics

Experiment No.	Cell Nos.	Test No.	Temperature	Separator Thickness	Separator Material
18a	F-399 to F-401	1a, b, c	+10°C	.010"	1 Layer Glass
	F-402 to F-404	2a, b, c	+10°C	.015"	1 Layer Glass
	F-405 to F-407	3a, b, c	+10°C	.020"	2 Layers Glass
	F-408 to F-410	4a, b, c	+40°C	.010"	1 Layer Glass
	F-411 to F-413	5a, b, c	+40°C	.015"	1 Layer Glass
	F-414 to F-416	6a, b, c	+40°C	.020"	2 Layers Glass
18c	F-417 to F-419	1a, b, c	+40°C	.008"	Porvic
	F-420 to F-422	2a, b, c	+40°C	.020"	Porvic
	F-423 to F-425	3a, b, c	+40°C	.008"	Porvic
				.010"	Glass

The remaining factors were constant for all cells:

Discharge Rate	10 ma/cm <sup>2</sup> at 3.0 volts
Electrolyte Molarity	2.1
Electrolyte Volume	45 cc
Cathode Thickness	.080"
Cathode Conductor Material	Asbury
Cathode Conductor Content	7%
Cathode Binder Content	1%
Cathode H <sub>2</sub> O Content	4%
Screen Geometry	Exmet 5Ag 14-1/0
Anode Capacity	150% of Cathode
Electrode Size	2" x 3"
No. of Electrodes	3 (1 positive and 2 negative)

TABLE 50

Experiment #18a and c - Discharge Data and Responses

Cell No.	Initial CCV	Max. CCV	Avg. Voltage		Amp-Hrs/Lb CuF <sub>2</sub>		Whrs/Lb CuF <sub>2</sub>		Whrs/Cm <sup>3</sup> Cell		
			2.5 v	2.0 v	2.5 v	2.0 v	2.5 v	2.0 v	2.5 v	2.0 v	
			<u>Experiment #18a</u>								
F-399	0.48	3.16	2.97	2.86	152	174	453	498	.792	.870	
F-400	0.27	3.12	2.94	2.88	168	190	495	548	.888	.984	
F-401 *	0.75	3.10	2.93	2.82	160	197	469	554	.849	1.003	
F-402	1.36	3.07	2.91	2.80	139	175	404	489	.666	.806	
F-403	0.28	3.12	2.98	2.92	178	195	530	568	.875	.937	
F-404	1.18	3.05	2.91	2.82	159	179	463	506	.761	.833	
F-405	0.26	3.16	2.99	2.91	172	192	514	559	.804	.874	
F-406	0.59	3.15	2.98	2.88	156	187	465	538	.721	.834	
F-407	0.28	3.11	2.92	2.82	146	173	425	488	.636	.729	
F-408	3.16	3.22	3.05	2.97	141	154	430	456	.769	.817	
F-409	3.12	3.24	3.03	2.91	155	179	470	522	.831	.921	
F-410	3.12	3.21	3.03	2.90	148	168	448	488	.791	.861	
F-411	3.12	3.19	3.00	2.85	119	141	357	401	.604	.678	
F-412	3.14	3.18	2.98	2.82	111	130	329	366	.551	.613	
F-413	3.16	3.21	3.07	2.93	149	169	—	456	496	.740	.804
F-414	3.15	3.21	3.07	2.98	170	181	523	538	.816	.840	
F-415	3.15	3.20	3.03	2.94	164	180	497	530	.791	.843	
F-416	3.16	3.25	3.11	3.08	183	193	569	569	.895	.936	

\* Environment failure at 5.62 hours, causing temperature to go to room ambient and thus affects values to 2.0 volts.

TABLE 50 (con't)

Experiment #18a and c - Discharge Data and Responses

Cell No.	Initial	Max.	Avg. Voltage		Amp-Hrs/Lb		Whrs/Lb CuF <sub>2</sub>		Whrs/Cm <sup>3</sup> Cell	
	CCV	CCV	2.5 v	2.0 v	CuF <sub>2</sub>		2.5 v	2.0 v	2.5 v	2.0 v
					2.5 v	2.0 v				
<u>Experiment #18c</u>										
F-417	3.03	3.10	2.87	2.68	94.0	125	270	337	.498	.621
F-418	3.09	3.16	2.95	2.81	109	132	322	371	.574	.661
F-419	2.22	3.00	2.79	2.64	15.0	18.6	41.9	49.3	.075	.088
F-420	2.95	2.95	2.78	2.68	46.7	56.4	130	151	.202	.235
F-421	3.04	3.04	2.85	2.70	108	142	306	383	.474	.593
F-422	2.94	3.00	2.82	2.66	105	141	294	377	.465	.594
F-423	3.02	3.16	3.02	2.95	166	178	500	526	.791	.832
F-424	3.04	3.16	3.00	2.97	180	194	539	576	.853	.912
F-425	3.04	3.16	3.02	2.95	177	187	534	552	.845	.872

TABLE 51

Experiment #25 Cell Construction Characteristics

Cell Nos.	Electrode Size	Test Vehicle	Active Stand (Days)	SO <sub>2</sub> Atmosphere	Separator Material (Glass)
<u>Experiment #25a</u>					
G-526, G-539	1½" x 5/8"	Glass Tube	0	No	2 Layers/.020" Thick
G-565 & G-566					
G-532 to G-534	1½" x 5/8"	Glass Tube	7	No	2 Layers/.020" Thick
G-567 to G-569	1½" x 5/8"	Glass Tube	0	Yes	2 Layers/.020" Thick
G-549 to G-551	1½" x 5/8"	Glass Tube	7	Yes	2 Layers/.020" Thick
<u>Experiment #25b</u>					
F-559 to F-561	2" x 3"	Std. NASA	7	Yes	2 Layers/.020" Thick
<u>Experiment #25c</u>					
G-555 to G-557	1½" x 5/8"	Glass Tube	0	Yes	2 Layers Glass (.020" Thick) /
					1 Layer Porvic (.008" Thick) *
G-558, G-570	1½" x 5/8"	Glass Tube	7	Yes	2 Layers Glass (.020" Thick) /
& G-571					1 Layer Porvic (.008" Thick) *

The remaining factors were constant for all cells:

Temperature	+40°C
Discharge Rate	10 ma/cm <sup>2</sup> at 3.0 volts
Electrolyte Volume	7.5 cc Glass Tube
	45 cc Std. NASA
Cathode Thickness	.050"
Cathode Conductor Material	Conductex SC
Cathode Conductor Content	21%
Cathode Binder Content	.5%
Cathode H <sub>2</sub> O Content	0%
Screen Geometry	Exmet 5Ag 14-1/0
Anode Capacity	150% of Cathode
No. of Electrodes	3

\* Porvic positioned between glass layers.

TABLE 52

Experiment #25 - Discharge Data and Responses

Cell No.	OCV	Initial	Max.	Avg. Voltage		Discharge		Amp-Hrs/Lb		Whrs/Lb		Whrs/Cm <sup>3</sup>	
		CCV	CCV	2.5 v	2.0 v	Life - Hours		CuF <sub>2</sub>		CuF <sub>2</sub>		Cell	
						2.5 v	2.0 v	2.5 v	2.0 v	2.5 v	2.0 v	2.5 v	2.0 v
<u>Experiment #25a</u>													
G-526	3.41	2.79	3.24	3.07	3.04	3.27	3.47	176	185	541	564	.554	.577
G-539	3.43	2.74	3.19	3.04	3.01	2.85	3.10	167	180	509	541	.460	.489
G-565	3.42	3.20	3.28	3.14	3.10	3.02	3.32	166	180	521	539	.454	.488
G-566	3.44	3.09	3.17	3.07	2.78	3.55	3.78	198	209	610	638	.525	.550
G-532	2.38	2.19	2.19	-	2.09	-	0.417	-	16.2	-	33.9	-	.028
G-533	3.12	2.58	2.58	2.55	2.28	0.02	0.983	0.8	39.8	1.9	90.6	.002	.084
G-534	3.00	2.39	2.39	-	2.18	-	0.550	-	20.9	-	45.7	-	.046
G-567	3.41	2.89	3.17	2.95	2.93	3.37	3.63	185	198	546	579	.466	.495
G-568	3.42	3.10	3.21	3.03	2.98	3.65	3.88	198	207	602	618	.543	.558
G-569	3.42	2.67	3.06	2.89	2.85	2.03	2.21	149	161	432	458	.269	.285
G-549	3.34	3.02	3.19	3.05	2.98	3.35	3.70	176	190	538	567	.559	.590
G-550	3.34	2.79	3.08	2.96	2.87	2.80	2.22	147	164	436	470	.446	.481
G-551	3.33	2.90	3.12	3.00	2.95	3.30	3.63	172	186	516	549	.503	.535
<u>Experiment #25b</u>													
G-559	2.97	2.22	2.32	2.89	2.12	0.02	0.10	1.0	4.2	2.8	9.0	.003	.009
G-560	3.35	2.64	2.66	2.62	2.41	0.55	1.20	27.8	55.9	72.8	134	.075	.138
G-561	3.12	2.66	2.69	2.64	2.46	0.28	0.55	15.1	27.3	39.7	67.2	.041	.070
<u>Experiment #25c</u>													
G-555	3.41	2.71	2.72	2.65	2.53	1.12	1.60	53.1	72.6	141	182	.121	.158
G-556	3.41	2.70	2.71	2.66	2.54	1.07	1.53	50.4	69.2	134	175	.121	.159
G-557	3.41	2.69	2.71	2.65	2.55	1.10	1.53	51.6	69.2	136	176	.171	.151
G-558	3.31	2.77	2.77	2.63	2.49	0.983	1.56	46.5	70.1	122	174	.106	.151
G-570	3.32	2.84	3.09	2.92	2.86	2.68	3.32	136	155	399	445	.338	.377
G-571	3.32	2.80	3.00	2.84	2.77	2.11	3.32	103	112	293	310	.261	.275

TABLE 53

Experiment #27 Cell Construction Characteristics

Cell Nos.	Stand	Anode Screen
G-572 to G-574	7 Days	SS 2/0
G-575 to G-577	14 Days	SS 2/0
G-578 to G-580	7 Days	Ni 3/0
G-581 to G-583	14 Days	Ni 3/0
G-584 to G-586	7 Days	Cu 2/0
G-587 to G-589	14 Days	Cu 2/0

The remaining factors were constant for all cells:

Temperature	+40°C
Discharge Rate	10 ma/cm <sup>2</sup> at 3.0 volts
Electrolyte Molarity	2.5
Electrolyte Volume	7.5 cc
Cathode Thickness	.050"
Cathode Conductor Material	Conductex SC
Cathode Conductor Content	21%
Cathode Binder Content	.5%
Cathode H <sub>2</sub> O Content	0%
Screen Geometry	Exmet 5Ag 14-1/0
Separator Material	Glass, 2 Layers - Long Wicks
Separator Thickness	.020"
Anode Capacity	>150% of Cathode
Electrode Size	5/8" x 1-1/2"
No. of Electrodes	3 (1 positive and 2 negative)
Atmosphere	SO <sub>2</sub> - 2 psi - Eq. at Room Temperature

TABLE 54

Experiment #27 - Discharge Data and Responses

Cell No.	OCV	Initial CCV	Max. CCV	Avg. Voltage		Discharge Life - Hours		Amp-Hrs/Lb CuF <sub>2</sub>		Whrs/Lb CuF <sub>2</sub>		Whrs/Cm <sup>3</sup> Cell	
				2.5 v	2.0 v	2.5 v	2.0 v	2.5 v	2.0 v	2.5 v	2.0 v	2.5 v	2.0 v
G-572	3.35	2.93	3.01	2.79	2.70	2.61	3.20	128	152	359	412	.305	.350
G-573	3.38	2.92	3.00	2.76	2.68	2.21	2.85	107	120	296	321	.252	.272
G-574	3.37	3.02	3.06	2.80	2.75	2.55	2.88	128	143	360	392	.294	.320
G-575	2.76	1.45	1.45	-	-	-	-	-	-	-	-	-	-
G-576	3.30	2.76	2.76	2.60	2.43	0.58	1.53	27.3	67.3	70.8	163	.059	.136
G-577	2.43	0.45	0.45	-	-	-	-	-	-	-	-	-	-
G-578	3.37	2.89	2.89	2.68	2.50	1.37	2.53	64.8	112	173	280	.146	.236
G-579	3.36	3.03	3.08	2.85	2.78	2.62	3.15	130	153	372	424	.331	.378
G-580	3.36	2.98	3.07	2.87	2.78	2.75	3.18	136	153	391	426	.348	.379
G-581	2.83	2.53	2.53	2.52	2.37	0.05	0.27	2.2	11.3	5.7	26.7	.004	.021
G-582	2.80	2.55	2.52	2.53	2.42	0.08	0.25	3.7	10.5	9.3	25.5	.008	.022
G-583	3.33	2.72	2.72	2.54	2.41	0.92	2.22	42.5	97.7	107	236	.089	.194
G-584	3.36	2.95	3.07	2.87	2.84	2.38	2.63	121	133	348	377	.278	.301
G-585	3.35	2.86	3.09	2.88	2.83	2.48	2.78	128	141	370	399	.321	.346
G-586	3.35	2.82	3.06	2.85	2.79	2.56	2.86	132	145	377	403	.323	.347
G-587	3.32	2.69	2.86	2.67	2.59	2.35	3.25	111	150	298	389	.245	.320
G-588	3.33	2.70	2.70	2.58	2.48	1.70	2.87	74.8	121	192	301	.171	.268
G-589	3.33	2.78	2.82	2.72	2.62	1.96	2.73	91.8	123	249	321	.226	.291

TABLE 55

Experiment #28 Cell Construction Characteristics

Cell No.	Temperature	Discharge Rate	Electrolyte Volume	Screen Geometry
F-590 to F-592	+40°C	10 ma/cm <sup>2</sup>	20 cc	Exmet 5Ag 14-1/0
F-593 to F-595	+40°C	10 ma/cm <sup>2</sup>	20 cc	Cathode - 5Ag 14-1/0--Anode - SS 3/0
F-596 to F-598	+40°C	10 ma/cm <sup>2</sup>	15 cc	Exmet 5Ag 14-1/0

The remaining factors were constant for all cells:

Electrolyte Molarity	2.5
Cathode Thickness	.050"
Cathode Conductor Material	Conductex SC
Cathode Conductor Content	21%
Cathode Binder Content	.5% Styrene
Cathode H <sub>2</sub> O Content	0%
Separator Material	Glass
Separator Thickness	2 Layers - .020"
Anode Capacity	150% of Cathode
Electrode Size	2" x 3"
No. of Electrodes	3



TABLE 56

Experiment #28 - Discharge Data and Responses

Cell No.	OCV	Initial CCV	Max. CCV	Avg. Voltage		Discharge Life - Hours		Amp-Hrs/Lb CuF <sub>2</sub>		Whrs/Lb CuF <sub>2</sub>		Whrs/Cm <sup>3</sup> Cell	
				2.5 v	2.0 v	2.5 v	2.0 v	2.5 v	2.0 v	2.5 v	2.0 v	2.5 v	2.0 v
F-590	3.44	2.94	3.27	3.13	3.09	3.05	3.17	197	201	617	623	.608	.614
F-591	3.43	2.76	3.26	3.14	3.10	2.65	2.78	165	171	518	533	.514	.528
F-592	3.43	2.67	2.85	2.77	2.75	2.10	2.32	132	144	366	398	.319	.346
F-593	3.42	2.62	3.14	3.01	2.99	3.27	3.48	195	206	587	616	.560	.587
F-594	3.42	2.53	3.08	2.93	2.90	3.02	3.27	178	191	524	557	.496	.527
F-595	3.42	2.57	2.68	2.63	2.62	1.92	2.15	114	128	301	335	.271	.302
F-596	3.44	2.70	3.22	3.06	2.95	2.15	3.07	168	187	515	552	.501	.537
F-597	3.43	2.63	2.66	2.62	2.47	0.90	1.62	47.8	80.9	125	200	.124	.198
F-598	3.43	2.71	2.84	2.78	2.76	2.42	2.62	145	156	405	431	.377	.402

TABLE 57

Experiment #29 Cell Construction Characteristics

Cell Nos.	SO <sub>2</sub> Quantity	Electrolyte Volume	Active Stand (Days)
F-599 to F-601	.135 g/g elect.	25 cc	0
F-602 to F-604	.135 g/g elect.	20 cc	0
F-605 to F-607	.090 g/g elect.	20 cc	7
F-608 to F-610	.090 g/g elect.	25 cc	7
F-611 to F-613	.090 g/g elect.	20 cc	14
F-614 to F-616	.090 g/g elect.	25 cc	14

The remaining factors were constant for all cells:

Temperature	+40 °C (Active stand and discharge.)
Discharge Rate	10 ma/cm <sup>2</sup> at 3.0 volts
Electrolyte Molarity	2.5
Cathode Thickness	.050"
Cathode Conductor Material	Conductex
Cathode Conductor Content	21%
Cathode Binder Content	.5%
Cathode H <sub>2</sub> O Content	0%
Screen Geometry	Anode - Exmet 5Cu 7-1/0 Cathode - Exmet 5Ag 14-1/0
Separator Material	Glass, 2 Layers
Separator Thickness	.020"
Electrode Size	2" x 3"
No. of Electrodes	3

TABLE 58

Experiment #29 - Discharge Data and Responses

Cell No.	OCV	Initial CCV	Max. CCV	Avg. Voltage		Discharge Life - Hours		Amp-Hrs/Lb CuF <sub>2</sub>		Whrs/Lb CuF <sub>2</sub>		Whrs/Cm <sup>3</sup> Cell	
				2.5 v	2.0 v	2.5 v	2.0 v	2.5 v	2.0 v	2.5 v	2.0 v	2.5 v	2.0 v
F-599	3.42	3.11	3.23	3.12	3.09	4.12	4.32	217	226	679	700	.718	.741
F-600	3.43	2.82	3.24	3.08	3.06	4.03	4.28	212	222	653	679	.714	.741
F-601	3.43	2.76	3.20	3.03	3.01	3.90	4.13	200	210	607	633	.639	.667
F-602	3.43	2.75	3.20	3.03	2.99	4.16	4.38	214	222	651	664	.666	.679
F-603	3.43	2.77	3.23	3.07	3.04	4.05	4.28	213	223	654	680	.720	.749
F-604	3.43	2.74	3.12	2.95	2.93	4.20	4.43	207	217	611	636	.685	.712
F-605	3.38	3.12	3.22	3.09	3.06	3.86	4.10	201	211	621	647	.653	.680
F-606	3.36	3.02	3.17	3.03	2.98	3.86	4.23	204	220	620	655	.621	.657
F-607	2.82	2.02	2.02	-	-	-	-	-	-	-	-	-	-
F-608	1.76	0.28	0.28	-	-	-	-	-	-	-	-	-	-
F-609	2.80	1.34	1.40	-	-	-	-	-	-	-	-	-	-
F-610	1.72	0.22	0.22	-	-	-	-	-	-	-	-	-	-
F-611	3.35	3.06	3.17	3.01	2.95	3.85	4.28	194	212	583	627	.596	.641
F-612	3.36	3.08	3.14	2.97	2.90	3.73	4.30	193	217	574	629	.602	.660
F-613	0.88	0.15	0.15	-	-	-	-	-	-	-	-	-	-
F-614	1.08	0.20	0.20	-	-	-	-	-	-	-	-	-	-
F-615	3.29	2.83	2.85	2.63	2.48	0.42	0.77	19.5	33.8	51.2	83.9	.051	.084
F-616	1.73	0.20	0.20	-	-	-	-	-	-	-	-	-	-

TABLE 59

Experiment #31 Cell Construction Characteristics

Cell Nos.	Storage Time (Days)	CuF <sub>2</sub> Source	Electrolyte Source
G-677 to G-679	0	Battelle	Honeywell *
G-680, G-681 & G-634	7	Battelle	Honeywell *
G-635 to G-637	14	Battelle	Honeywell *
G-638 to G-640	0	Battelle	U. S. Steel
G-641 to G-643	7	Battelle	U. S. Steel
G-644 to G-646	14	Battelle	U. S. Steel
G-647 to G-649	0	Battelle	MRI
G-650 to G-652	7	Battelle	MRI
G-653 to G-655	14	Battelle	MRI
G-659 to G-661	0	Ozark	U. S. Steel
G-662 to G-664	7	Ozark	U. S. Steel
G-665 to G-667	14	Ozark	U. S. Steel
G-668 to G-670	0	Ozark	MRI
G-671 to G-673	7	Ozark	MRI
G-674 to G-676	14	Ozark	MRI

The remaining factors were constant for all cells:

Discharge Temperature	+40°C
Storage Temperature	+40°C
Discharge Rate	10 ma/cm <sup>2</sup> at 3.0 volts
Electrolyte Molarity	2.5 - Wicked Activation
Electrolyte Volume	7.5 cc
Cathode Thickness	.050"
Cathode Conductor Material	Conductex SC
Cathode Conductor Content	21%
Cathode Binder Content	.5%
Cathode H <sub>2</sub> O Content	0%
Screen Geometry	Anode - Exmet 5Cu 7-1/0 Cathode - Exmet 5Ag 14-1/0
Separator Thickness	.020" - 2 Layers
Electrode Size	1-1/2" x 5/8"
No. of Electrodes	3
SO <sub>2</sub> Quantity	0.113 g SO <sub>2</sub> per gram Electrolyte

\* Metathetically prepared by Honeywell from Ozark KAsF<sub>6</sub> and Foote Mineral LiBF<sub>4</sub>

TABLE 60

Experiment #31 - Discharge Data and Responses

Cell No.	OCV	Initial	Max.	Avg. Voltage		Discharge		Amp-Hrs/Lb		Whrs/Lb CuF <sub>2</sub>		Whrs/Cm <sup>3</sup>	
		CCV	CCV	2.5 v	2.0 v	Life - Hours		CuF <sub>2</sub>		2.5 v	2.0 v	2.5 v	2.0 v
G-647	3.39	2.74	2.77	2.70	2.54	2.10	3.33	95.4	142.0	257.0	361.0	.230	.323
G-648	3.40	2.73	2.77	2.70	2.52	1.95	3.01	90.0	129.0	242.0	326.0	.213	.286
G-649	3.42	2.79	2.79	2.71	2.56	2.30	3.43	104.0	146.0	281.0	376.0	.255	.340
G-650	3.19	2.70	2.70	2.67	2.47	1.32	2.65	60.8	113.0	162.0	279.0	.146	.252
G-651	3.25	2.70	2.71	2.67	2.41	1.48	2.95	67.3	123.0	179.0	302.0	.160	.269
G-652	3.22	2.69	2.69	2.66	2.44	1.32	2.78	59.5	115.0	158.0	282.0	.143	.256
G-653	3.23	2.71	2.71	2.59	2.33	0.45	1.97	19.5	76.9	50.5	179.0	.045	.160
G-654	3.12	2.65	2.65	2.58	2.41	0.90	2.33	41.0	99.2	105.0	239.0	.088	.200
G-655	3.14	2.65	2.65	2.60	2.36	0.40	1.65	18.2	68.1	47.2	160.0	.041	.138
G-659	3.44	2.90	3.21	3.03	2.98	4.12	4.35	214.0	222.0	647.0	661.0	.591	.607
G-660	3.42	3.04	3.19	3.00	2.98	3.75	3.98	197.0	208.0	591.0	618.0	.523	.547
G-661	3.44	2.94	3.22	3.05	3.02	4.03	4.25	215.0	225.0	656.0	680.0	.575	.596
G-662	2.67	1.90	1.90	-	-	-	-	-	-	-	-	-	-
G-663	2.81	2.48	2.48	-	2.31	-	0.08	-	3.4	-	7.8	-	.007
G-664	3.30	2.89	2.89	2.61	2.42	0.52	1.28	23.7	54.6	61.9	131.0	.054	.115
G-665	2.77	2.10	2.10	-	2.05	-	0.02	-	0.6	-	1.2	-	.001
G-666	2.82	2.43	2.43	-	2.23	-	0.15	-	5.9	-	13.1	-	.012
G-667	1.75	0.71	0.71	-	-	-	-	-	-	-	-	-	-
G-668	3.43	2.82	2.82	2.69	2.60	1.45	1.87	68.9	85.6	185.0	222.0	.162	.195
G-669	3.43	2.78	2.78	2.69	2.62	1.47	1.82	70.6	85.2	189.0	223.0	.165	.194
G-670	3.42	2.71	2.71	2.64	2.53	1.08	1.60	51.0	72.0	134.0	182.0	.118	.160
G-671	3.26	2.68	2.68	2.59	2.40	0.53	1.57	24.2	66.1	62.7	158.0	.053	.135

TABLE 60 (con't)

Experiment #31 - Discharge Data and Responses

Cell No.	OCV	Initial CCV	Max. CCV	Avg. Voltage		Discharge Life - Hours		Amp-Hrs/Lb CuF <sub>2</sub>		Whrs/Lb CuF <sub>2</sub>		Whrs/Cm <sup>3</sup> Cell	
				2.5 v	2.0 v	2.5 v	2.0 v	2.5 v	2.0 v	2.5 v	2.0 v	2.5 v	2.0 v
G-672	3.19	2.70	2.70	2.64	2.47	0.38	0.78	17.9	34.1	47.2	84.3	.042	.075
G-673	3.21	2.68	2.68	2.62	2.40	0.38	1.03	17.8	44.1	46.7	106.0	.039	.089
G-674	3.11	2.66	2.66	2.61	2.43	0.48	1.67	22.0	49.6	57.4	120.0	.051	.107
G-675	3.05	2.67	2.68	2.63	2.47	0.37	0.70	16.7	29.9	44.0	73.9	.039	.066
G-676	3.07	2.67	2.67	2.61	2.45	0.42	0.87	19.5	38.1	51.0	93.2	.043	.079
G-677	3.42	3.23	3.24	3.05	3.00	3.47	3.78	182.0	195.0	555.0	584.0	.526	.553
G-678	3.42	3.22	3.25	3.06	3.00	3.85	4.30	200.0	219.0	611.0	658.0	.565	.608
G-679	3.43	3.23	3.24	2.98	2.90	3.32	3.88	166.0	189.0	496.0	549.0	.465	.515
G-680	3.36	3.04	3.16	2.93	2.85	2.83	3.30	139.0	157.0	408.0	449.0	.379	.416
G-681	3.31	3.03	3.20	2.96	2.89	2.68	3.15	133.0	153.0	395.0	443.0	.375	.421
G-634	3.34	2.76	3.00	2.81	2.74	3.10	3.60	146.0	166.0	411.0	455.0	.364	.403
G-635	3.32	2.91	3.03	2.81	2.68	2.11	2.73	104.0	128.0	293.0	345.0	.242	.285
G-636	3.32	2.98	3.08	2.83	2.71	2.26	2.88	110.0	134.0	314.0	365.0	.267	.310
G-637	3.33	2.97	3.04	2.83	2.72	2.33	2.97	110.0	135.0	313.0	368.0	.275	.323
G-638	3.41	2.84	2.84	2.73	2.64	2.52	3.32	122.0	155.0	334.0	410.0	.275	.338
G-639	3.41	2.82	2.85	2.75	2.65	2.96	3.75	146.0	178.0	404.0	474.0	.331	.388
G-640	3.43	2.86	2.94	2.77	2.64	2.93	3.88	139.0	176.0	386.0	465.0	.340	.410
G-641	2.89	2.67	2.67	2.60	2.41	0.43	0.93	19.7	39.2	51.3	94.4	.043	.080
G-642	2.85	2.67	2.67	2.60	2.53	0.32	0.48	13.9	20.6	36.2	51.9	.032	.046
G-643	2.93	2.67	2.67	2.59	2.44	0.45	0.85	20.7	36.9	53.7	90.0	.047	.078
G-644	2.78	2.48	2.48	-	2.31	-	0.22	-	8.7	-	20.2	-	.018
G-645	2.78	2.15	2.15	-	2.09	-	0.05	-	1.8	-	3.8	-	.003
G-646	2.83	2.64	2.64	2.59	2.46	0.18	0.42	8.2	17.7	21.2	43.6	.019	.040

TABLE 61

Experiment #30 Cell Construction Characteristics

Cell Nos.	SO <sub>2</sub> Quantity	Storage Temperature	Active Stand (Days)
G-620 to G-622	.09 g/g elect.	+40°C	7
G-623 to G-625	.09 g/g elect.	+40°C	14
G-626 to G-628	.09 g/g elect.	+20°C	14
G-656 to G-658	.135 g/g elect.	+40°C	7

The remaining factors were constant for all cells:

Discharge Temperature	+40°C
Discharge Rate	10 ma/cm <sup>2</sup> at 3.0 volts
Electrolyte Molarity	2.5 - Wicked Activation
Cathode Thickness	.050"
Cathode Conductor Material	Conductex SC
Cathode Conductor Content	21%
Cathode Binder Content	.5%
Cathode H <sub>2</sub> O Content	0%
Screen Geometry	Anode - Exmet - 5Cu 7-1/0 Cathode - Exmet - 5Ag 14-1/0
Separator Material	Glass, 2 Layers
Separator Thickness	.020"
Anode Capacity	150% of Cathode
Electrode Size	1-1/2" x 5/8"
No. of Electrodes	3

TABLE 62

Experiment #30 - Discharge Data and Responses

Cell No.	OCV	Initial CCV	Max. CCV	Avg. Voltage		Discharge Life - Hours		Amp-Hrs/Lb CuF <sub>2</sub>		Whrs/Lb CuF <sub>2</sub>		Whrs/Cm <sup>3</sup> Cell	
				2.5 v	2.0 v	2.5 v	2.0 v	2.5 v	2.0 v	2.5 v	2.0 v	2.5 v	2.0 v
G-620	3.34	2.70	2.70	2.64	2.54	1.02	1.57	47.4	70.1	125.0	178	.113	.160
G-621	3.25	2.69	2.69	2.63	2.43	0.57	1.18	25.7	49.6	67.7	121	.059	.105
G-622	3.31	2.62	2.62	2.58	2.43	0.50	1.30	22.9	56.1	59.0	136	.048	.110
G-623	3.31	2.62	2.92	2.75	2.71	2.73	3.08	127.0	141.0	350.0	383	.322	.352
G-624	3.26	2.55	2.55	2.54	2.30	0.22	1.45	9.4	57.1	23.9	131	.021	.116
G-625	3.31	2.64	2.65	2.61	2.53	1.45	2.03	66.6	90.7	174.0	230	.156	.206
G-626	3.28	2.64	2.65	2.62	2.48	0.90	1.53	39.9	64.5	105.0	160	.095	.146
G-627	3.33	2.63	2.63	2.60	2.47	0.87	1.53	39.8	66.9	104.0	165	.091	.146
G-628	3.35	2.64	2.64	2.61	2.47	0.82	1.47	37.4	63.6	97.5	157	.084	.135
G-656	3.35	2.70	3.12	2.95	2.89	3.37	3.68	169.0	182.0	500.0	525	.448	.471
G-657	3.15	2.64	2.64	2.57	2.39	0.43	1.22	19.4	50.8	49.9	122	.046	.112
G-658	3.30	2.61	2.62	2.58	2.48	1.05	1.78	49.7	81.2	128.0	201	.109	.172



TABLE 63

Experiment #33 Cell Construction Characteristics

<u>Cell Nos.</u>	<u>SO<sub>2</sub> Quantity</u>
G-694 to G-696	.090 g/g electrolyte
G-697 to G-699	.045 g/g electrolyte
G-700 to G-702	.023 g/g electrolyte

The remaining factors were constant for all cells:

Discharge Temperature	+40°C
Storage Temperature	+40°C
Discharge Rate	10 ma/cm <sup>2</sup> at 3.0 volts
Storage Time	14 Days
Electrolyte Molarity	2.5 LiAsF <sub>6</sub> /MF - Wicked Activation
Electrolyte Volume	7.5 cc (Age - 8 Days)
Cathode Thickness	.050"
Cathode Conductor Material	Conductex SC
Cathode Conductor Content	21%
Cathode Binder Content	.5%
Cathode H <sub>2</sub> O Content	0%
Screen Geometry	Anode - Exmet 5Cu 7-1/0 Cathode - Exmet 5Ag 14-1/0
Separator Material	Glass, 2 Layers
Separator Thickness	.020"
Electrode Size	1-1/2" x 5/8"
No. of Electrodes	3

TABLE 64

Experiment #33 - Discharge Data and Responses

Cell No.	OCV	Initial CCV	Max. CCV	Avg. Voltage		Discharge Life - Hours		Amp-Hrs/Lb CuF <sub>2</sub>		Whrs/Lb CuF <sub>2</sub>		Whrs/Cm <sup>3</sup> Cell	
				2.5 v	2.0 v	2.5 v	2.0 v	2.5 v	2.0 v	2.5 v	2.0 v	2.5 v	2.0 v
G-694	3.33	2.85	2.87	2.76	2.68	1.80	2.37	89.1	113.0	245	304.0	.211	.261
G-695	3.31	2.61	2.67	2.62	2.41	0.82	1.92	39.5	85.4	103	206.0	.088	.175
G-696	2.75	2.30	2.30	-	2.17	-	0.13	-	5.1	-	11.1	-	.010
G-697	2.70	1.45	1.45	-	-	-	-	-	-	-	-	-	-
G-698	2.75	1.71	1.71	-	-	-	-	-	-	-	-	-	-
G-699	2.74	1.91	1.91	-	-	-	-	-	-	-	-	-	-
G-700	2.39	1.23	1.23	-	-	-	-	-	-	-	-	-	-
G-701	2.69	1.78	1.78	-	-	-	-	-	-	-	-	-	-
G-702	2.74	2.39	2.39 *	-	-	-	-	-	-	-	-	-	-

\* Less than one minute above 2.0 volts.

TABLE 65

Experiment #32 Cell Construction Characteristics

Cell Nos.	Electrolyte	Electrolyte Age (At Time of Activation)
G-682 to G-684	Honeywell B41a *	115 Days
G-685 to G-687	Honeywell B44 *	32 Days
G-688 to G-690	Honeywell B45 *	9 Days
G-691 to G-693	U. S. Steel	23 Days

The remaining factors were constant for all cells:

Discharge Temperature	+40°C
Storage Temperature	+40°C
Discharge Rate	10 ma/cm <sup>2</sup> at 3.0 volts
Storage Time	14 Days
Electrolyte Molarity	2.5 LiAsF <sub>6</sub> /MF - Wicked Activation
Electrolyte Volume	7.5 cc
Cathode Thickness	.050"
Cathode Conductor Material	Conductex SC
Cathode Conductor Content	21%
Cathode Binder Content	.5%
Cathode H <sub>2</sub> O Content	0%
Screen Geometry	Anode - Exmet 5Cu 7-1/0 Cathode - Exmet 5Ag 14-1/0
Separator Material	Glass, 2 Layers
Separator Thickness	.020"
Electrode Size	1-1/2" x 5/8"
No. of Electrodes	3
SO <sub>2</sub> Quantity	0.113 g SO <sub>2</sub> /g electrolyte

\* Prepared at Honeywell metathetically from Ozark-Mahoning KAsF<sub>6</sub> and Foote Mineral LiBF<sub>4</sub>.

TABLE 66

Experiment #32 - Discharge Data and Responses

Cell No.	OCV	Initial CCV	Max. CCV	Avg. Voltage		Discharge Life - Hours		Amp-Hrs/Lb CuF <sub>2</sub>		Whrs/Lb CuF <sub>2</sub>		Whrs/Cm <sup>3</sup> Cell	
				2.5 v	2.0 v	2.5 v	2.0 v	2.5 v	2.0 v	2.5 v	2.0 v	2.5 v	2.0 v
G-682	3.32	2.81	3.07	2.93	2.89	2.50	2.73	139.0	150.0	407.0	434.0	.344	.367
G-683	3.33	2.63	2.66	2.61	2.39	0.88	2.83	39.4	118.0	102.0	283.0	.091	.249
G-684	3.32	2.75	2.75	2.61	2.49	0.60	1.15	28.9	52.8	75.4	131.0	.063	.110
G-685	3.32	2.86	3.04	2.86	2.76	1.53	1.88	81.5	96.7	232.0	267.0	.196	.225
G-686	2.82	2.68	2.68	2.62	2.49	0.32	0.50	14.5	21.8	38.1	54.3	.035	.050
G-687	3.28	2.57	2.57	2.52	2.36	0.32	2.40	13.7	97.0	34.4	228.0	.031	.209
G-688	2.81	2.28	2.28	-	2.16	-	0.08	-	3.1	-	6.6	-	.006
G-689	2.83	2.61	2.61	2.56	2.42	0.12	0.35	5.1	14.6	13.2	35.4	.012	.032
G-690	3.32	2.94	2.94	2.77	2.70	1.12	1.38	56.8	65.8	157.0	177.0	.133	.150
G-691	2.86	2.64	2.64	2.60	2.42	0.07	0.17	3.2	7.4	8.2	17.8	.007	.015
G-692	2.76	0.98	0.98	-	-	-	-	-	-	-	-	-	-
G-693	2.86	2.66	2.66	2.60	2.47	0.15	0.32	7.2	14.4	18.7	35.5	.016	.031

TABLE 67

Experiment #5 Cell Construction Characteristics

Cell No.	Test No.	Temp.	Discharge Rate ma/cm <sup>2</sup>	Separator * Material	Separator ** Thickness
F-117	1	+31 °C	13	A	.005"
F-118	2	+43 °C	13	A	.010"
F-119	3	+31 °C	19	A	.010"
F-120	4	+43 °C	19	A	.005"
F-121	5	+31 °C	13	B	.010"
F-122	6	+43 °C	13	B	.005"
F-123	7	+31 °C	19	B	.005"
F-124	8	+43 °C	19	B	.010"
F-125	9	Replicate Test #3			
F-126	10	Replicate Test #6			
F-128	11	Replicate Test #6			

The remaining factors were constant for all cells:

Electrolyte Molarity	2.0
Electrolyte Volume	110% Full
Cathode Thickness	.060"
Cathode Conductor Material	Asbury Ceylon Graphite
Cathode Conductor Content	10%
Cathode Binder Content	2%
Cathode H <sub>2</sub> O Content	8%
Screen Geometry	Standard Exmet 5Ag 14-1/0
Anode Capacity	125% of Cathode
Electrode Size	2-3/4" x 4"
No. of Electrodes	3 (1 positive and 2 negative)

\* Separator Material A = Webril Dacron E1486

B = Webloy Rayon Polypropylene Carded E4208

\*\* .005" thickness achieved using one layer of material per cathode/anode interface.

.010" thickness achieved using two layers of material per cathode/anode interface.

TABLE 68

Experiment #5 - Discharge Data and Responses

Cell No.	Temp.	Discharge Rate at 3.0 v	Max. CCV	Average Volts to 2.0 v	Y <sub>2</sub> Amp-Hrs/Lb CuF <sub>2</sub> to 2.0 v	Y <sub>4</sub> Watt-Hrs/Lb to 2.0 v	Y <sub>6</sub> Watt-Hrs/Cm <sup>3</sup> to 2.0 v
F-117	+31°C	13 ma/cm <sup>2</sup>	2.78	2.56	139.0	67.0	.561
F-118	+43°C	13 ma/cm <sup>2</sup>	2.50	2.27	100.0	43.4	.325
F-119	+31°C	19 ma/cm <sup>2</sup>	2.19	2.10	1.9	0.73	.005
F-120	+43°C	19 ma/cm <sup>2</sup>	2.50	2.37	111.0	51.3	.389
F-121	+31°C	13 ma/cm <sup>2</sup>	2.99	2.79	175.0	95.0	.668
F-122	+43°C	13 ma/cm <sup>2</sup>	3.08	2.85	174.0	99.1	.738
F-123	+31°C	19 ma/cm <sup>2</sup>	2.87	2.62	140.0	64.5	.495
F-124	+43°C	19 ma/cm <sup>2</sup>	2.87	2.69	157.0	77.8	.570
F-125	+31°C	19 ma/cm <sup>2</sup>	2.31	2.05	66.5	23.9	.180
F-126	+43°C	13 ma/cm <sup>2</sup>	3.07	2.98	113.0	60.4	.464
F-128	+43°C	13 ma/cm <sup>2</sup>	3.03	1.22	70.5	38.2	.305

TABLE 69

Experiment #5 Mean-Effect Levels - Yates Method

<u><math>y_2 = \text{Amp-Hrs/Lb of CuF}_2</math> to 2.0 v</u>		<u><math>y_4 = \text{Watt-Hrs/Lb to 2.0 v}</math></u>		<u><math>y_6 = \text{Watt-Hrs/Cm}^3 \text{ to 2.0 v}</math></u>	
Factor	Mean-Effect	Factor	Mean-Effect	Factor	Mean-Effect x 100
AB	+54.01	AB	+30.40	AB	+22.13
C	+44.89	C	+28.15	C	+19.15
BC	+31.01	B	-12.20	BC	+10.28
AC	-26.01	AC	-11.95	B	- 9.95
B	-15.89	BC	+11.20	AC	- 6.58
A	- 7.11	A	- 4.25	D	- 4.50
D	- 3.89	D	- 0.90	A	- 3.55

Temperature = A

Discharge Rate = B

Separator Material = C

Separator Thickness = D

AB (Confounded with CD)

BC (Confounded with AD)

AC (Confounded with BC)

} Interactions

TABLE 70

GC Analysis of Cell Discharge Gases

		Molecular Sieve Analysis						Chromosorb 102 Analysis							
Sample I. D.		Exp. No.	H <sub>2</sub>	O <sub>2</sub> -A <sub>r</sub> *	N <sub>2</sub>	CH <sub>4</sub>	CO	CO <sub>2</sub> and/ or CH <sub>3</sub> F	H <sub>2</sub> O	MeOH	MF	Unknown **	HCHO	SO <sub>2</sub>	CH <sub>3</sub> OH- CH <sub>3</sub> OCH <sub>3</sub>
No.															
F-108	4	3%	20%	37%	1%	-	-	-	2%	<1%	36%	-	-	-	-
F-109	4	2%	14%	27%	1%	-	-	-	1%	<1%	55%	-	-	-	-
F-112	4	2%	22%	24%	1%	-	-	-	1%	<1%	49%	-	-	-	-
F-120	5	3%	29%	24%	2%	<1%	-	-	1%	<1%	36%	5%	-	-	-
F-122	5	2%	16%	25%	1%	-	-	-	1%	1%	52%	2%	-	-	-
F-123	5	3%	26%	25%	2%	-	-	-	1%	<1%	40%	2%	-	-	-
F-137	6	1%	41%	23%	1%	-	-	-	1%	<1%	32%	-	-	-	-
F-140	6	2%	47%	19%	<1%	-	-	-	1%	<1%	31%	-	-	-	-
F-265	14d	1%	45%	18%	2%	<1%	-	-	1%	-	34%	<1%	-	-	-
F-269	14d	1%	72%	13%	5%	1%	-	-	1%	-	8%	<1%	-	-	-
F-272	14d	1%	69%	13%	4%	<1%	-	-	1%	-	12%	<1%	-	-	-
F-280	15a	1%	75%	14%	<1%	-	-	-	4%	-	5%	<1%	-	-	-
F-286	15a	2%	34%	22%	1%	<1%	-	-	5%	-	35%	1%	-	-	-
F-293	15a	1%	76%	14%	<1%	-	-	-	3%	-	6%	-	-	-	-
F-329	15b	1%	67%	12%	<1%	-	-	-	3%	<1%	17%	-	-	-	-
F-336	15b	1%	64%	11%	<1%	-	-	-	2%	-	22%	-	-	-	-
F-331	15b	1%	70%	11%	3%	<1%	-	-	2%	-	13%	-	-	-	-
F-375	15d	1%	52%	12%	Trace	-	-	-	1%	<1%	33%	-	-	-	-
F-379	15d	4%	42%	25%	1%	-	-	-	4%	<1%	23%	-	-	-	-
F-382	15d	1%	50%	10%	-	-	-	-	1%	-	38%	-	-	-	-
F-397	18b	1%	23%	5%	-	-	-	-	<1%	-	70%	-	2%	-	-
F-417	18c	3%	43%	25%	3%	<1%	-	-	2%	-	24%	-	-	-	-
F-425	18c	2%	16%	12%	4%	19%	<1%	-	1%	-	44%	-	2%	-	-
F-430	19a	2%	62%	18%	1%	-	-	-	2%	-	15%	-	<1%	-	-
F-433	19a	1%	62%	17%	<1%	-	-	-	1%	-	17%	-	1%	-	-
F-436	19a	2%	58%	23%	7%	1%	<1%	-	3%	-	4%	-	1%	-	-
F-524	26	<1%	50%	15%	1%	3%	-	-	1%	-	30%	-	-	-	<1%
F-526	25a	<1%	7%	23%	3%	8%	-	-	1%	-	57%	-	-	-	<1%
F-529	25a	1%	4%	16%	-	<1%	1%	<1%	-	-	23%	-	-	54%	-

\* Under conditions used O<sub>2</sub>-A<sub>R</sub> do not separate.

\*\* Tentatively identified as formaldehyde.

Note: Values given are the percent compound peak area of total peak area, and also the average of two separate determinations.



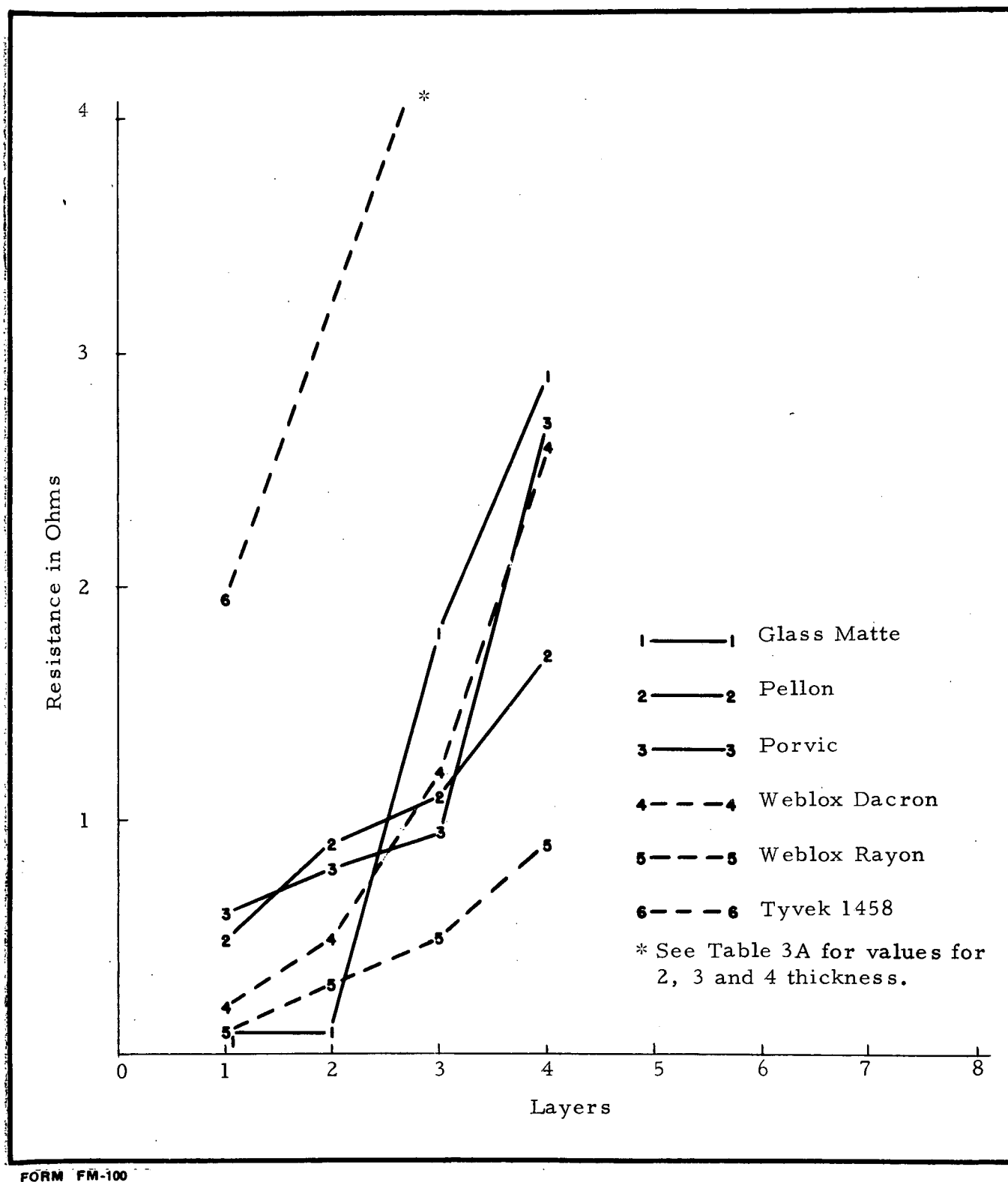


FIGURE 1

Resistance of Multiple Layer Separators - Initial Series

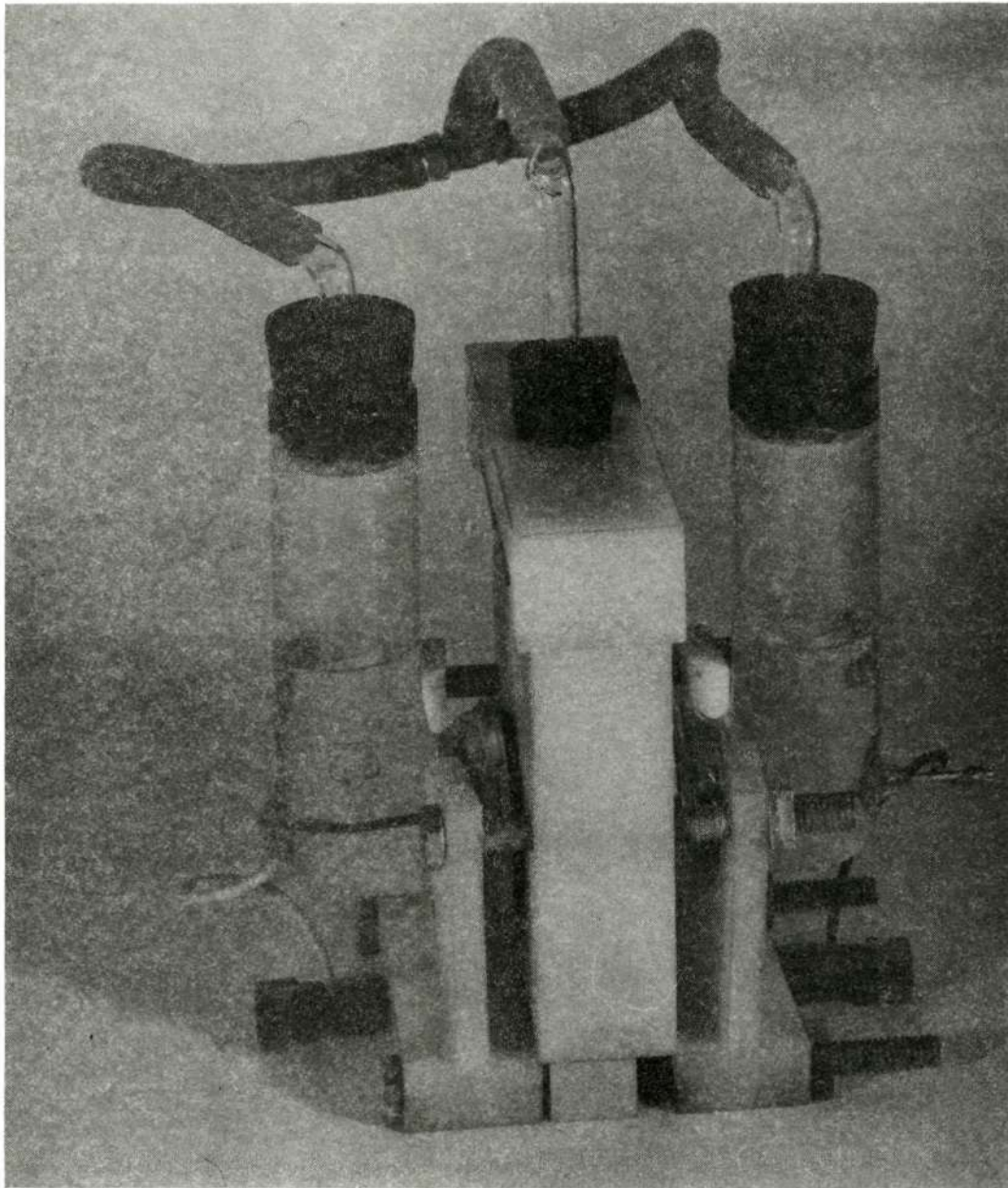
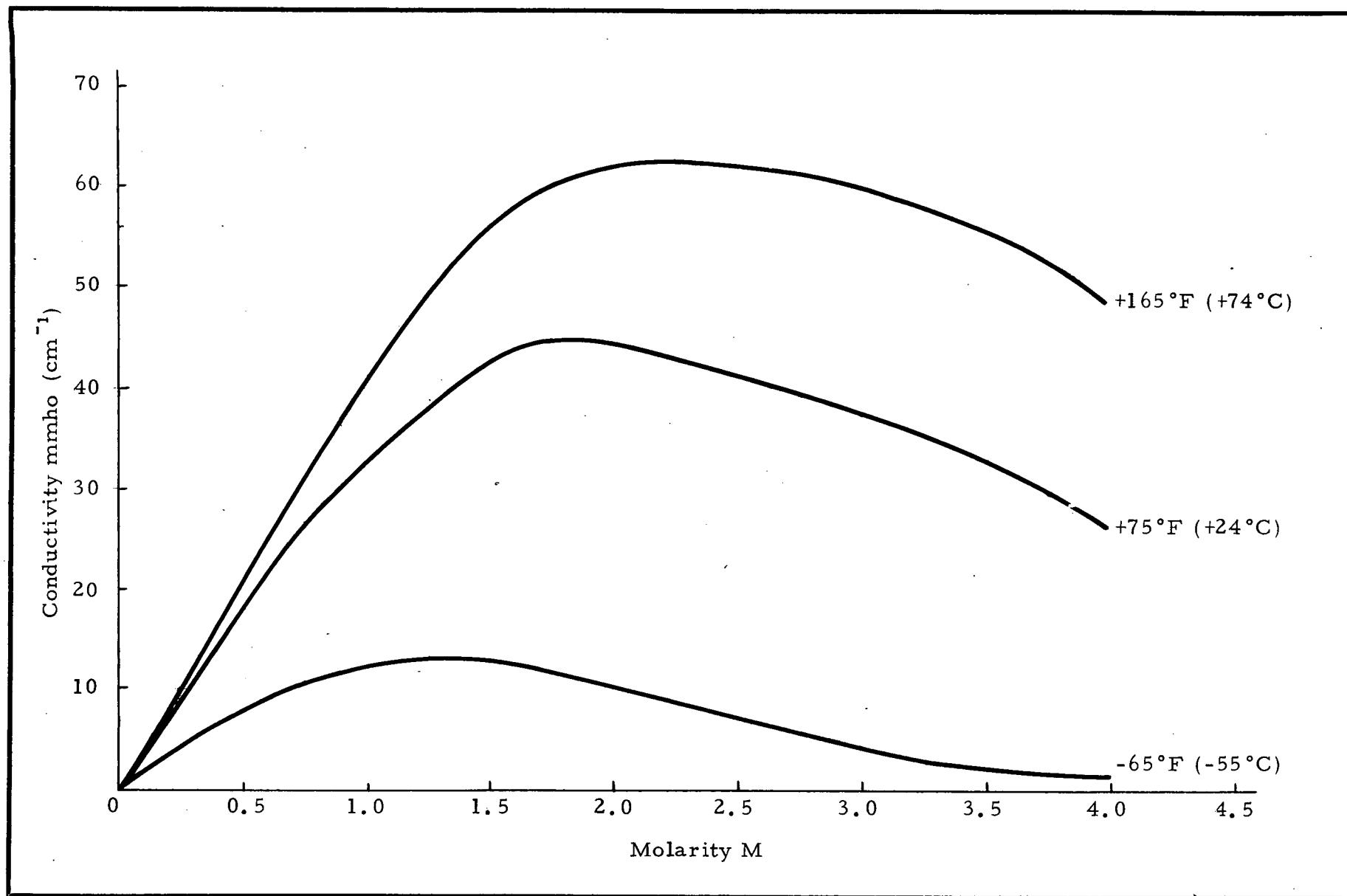


FIGURE 2

Resistance Test Fixture

This page is reproduced again at the back of this report by a different reproduction method so as to furnish the best possible detail to the user.



FORM FM-101

FIGURE 3

Conductivity Versus Molarity of LiAsF<sub>6</sub>/MF

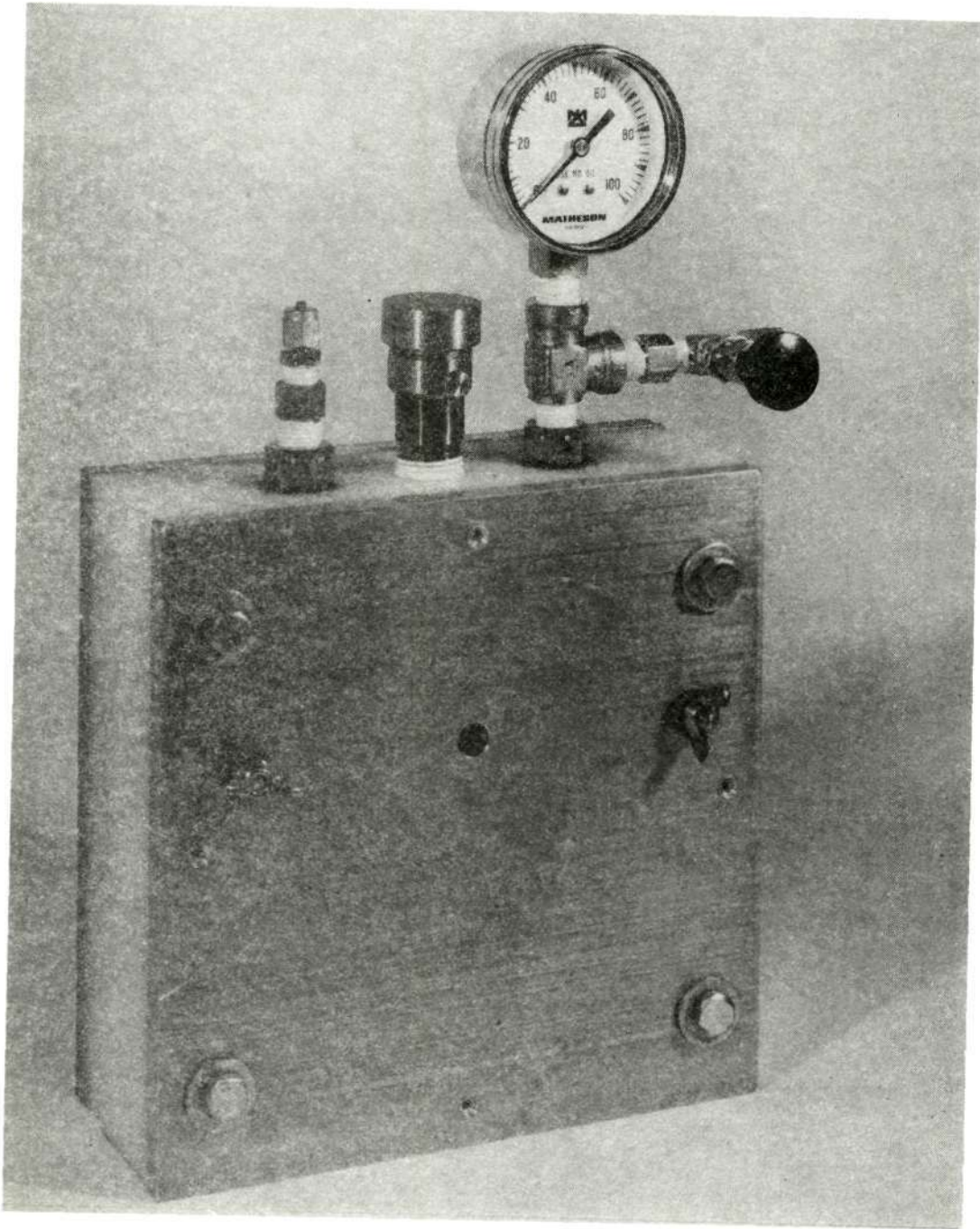


FIGURE 4

Cell Test Chamber

This page is reproduced again at the back of this report by a different reproduction method so as to furnish the best possible detail to the user.



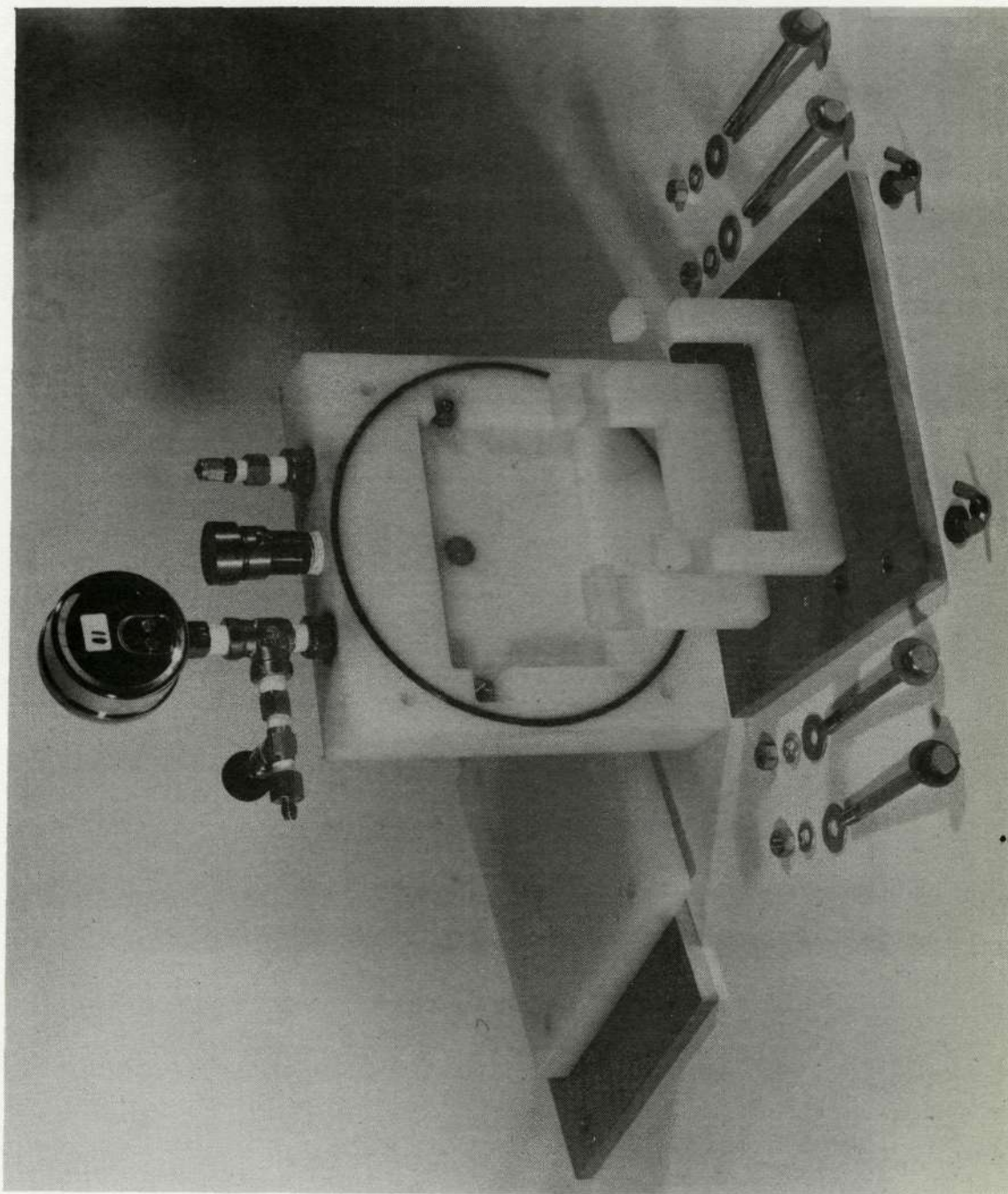


FIGURE 5  
Cell Test Fixture

This page is reproduced again at the back of this report by a different reproduction method so as to furnish the best possible detail to the user.

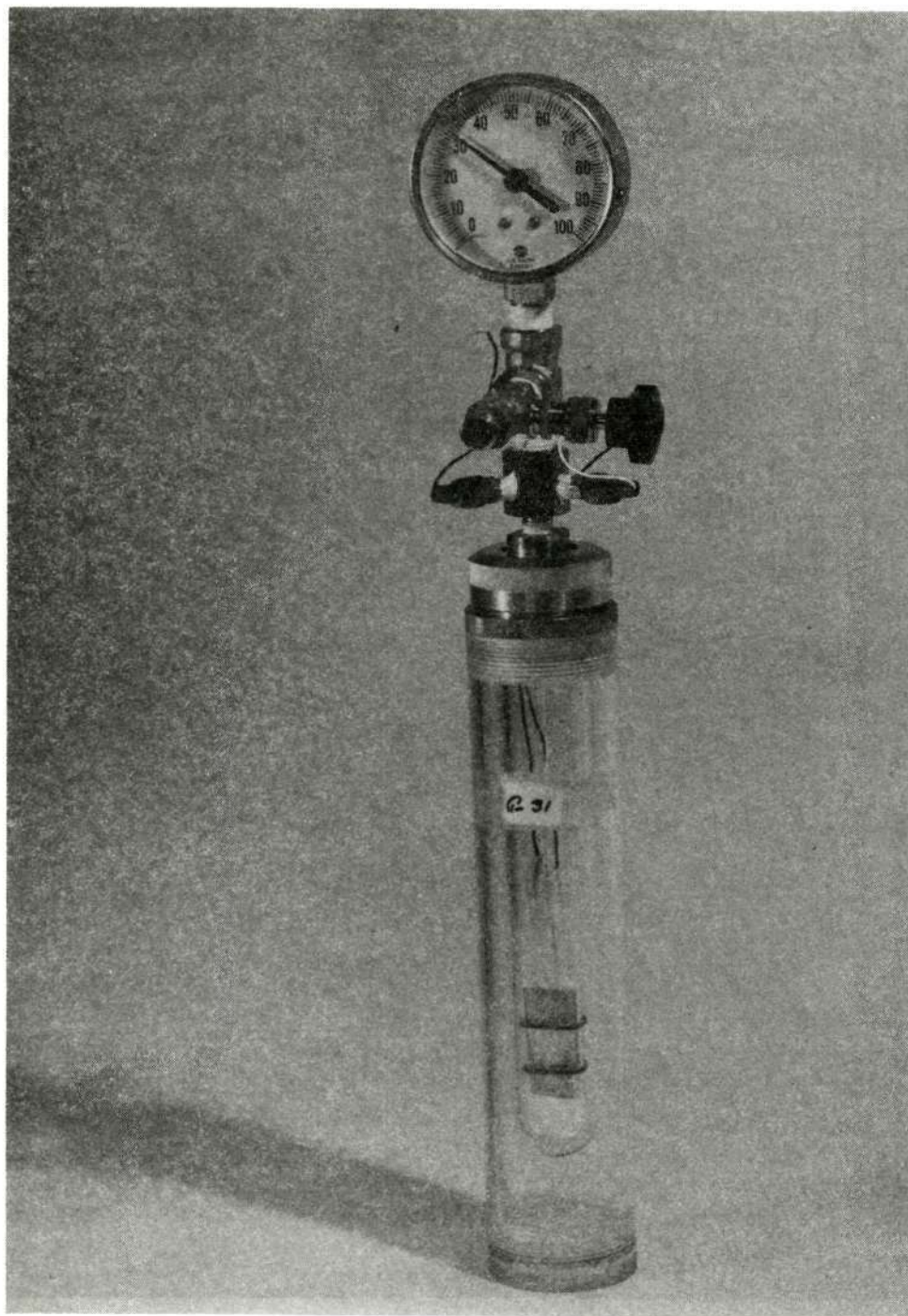


FIGURE 6

Glass Tube Cell Test Chamber

This page is reproduced again at the back of this report by a different reproduction method so as to furnish the best possible detail to the user.



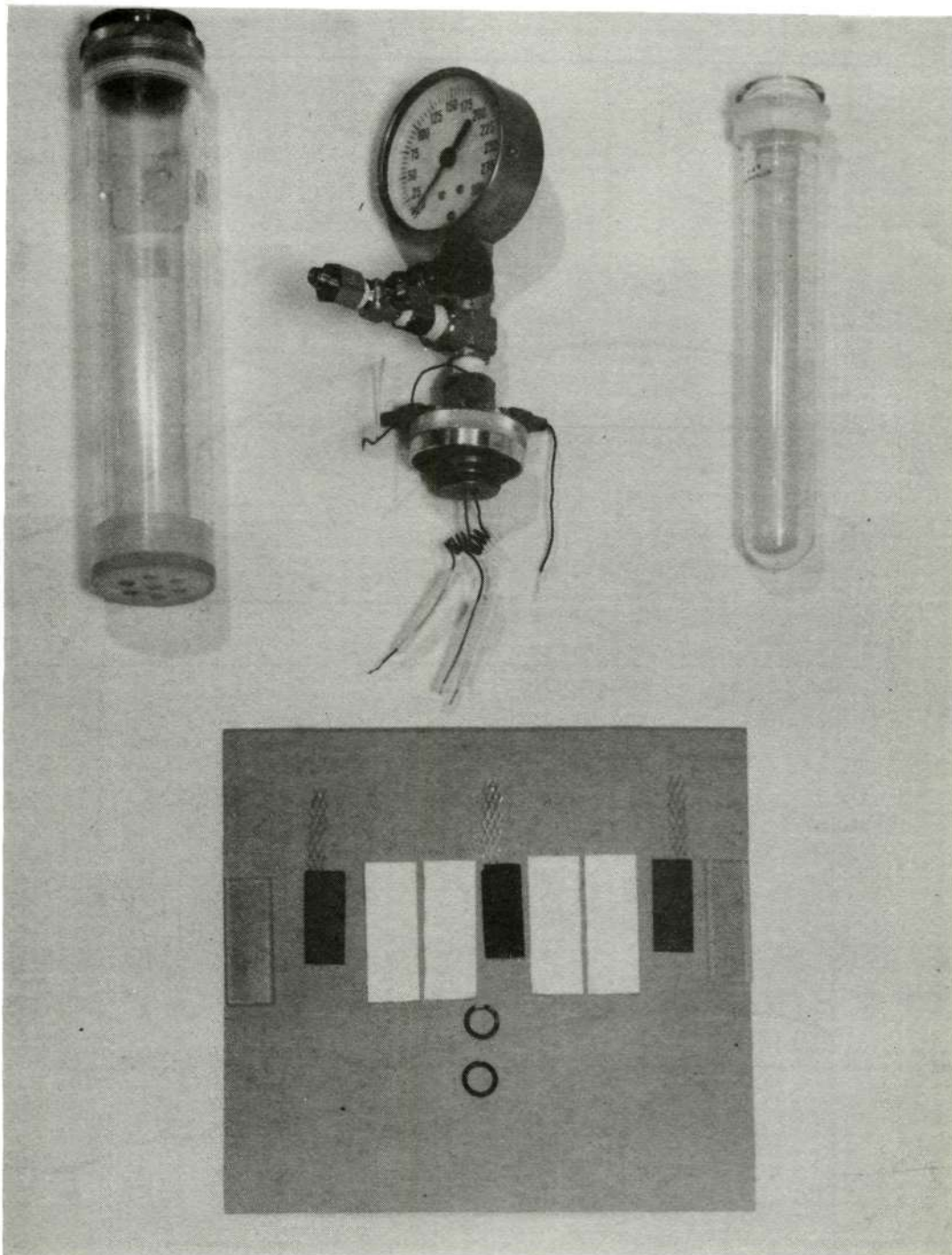
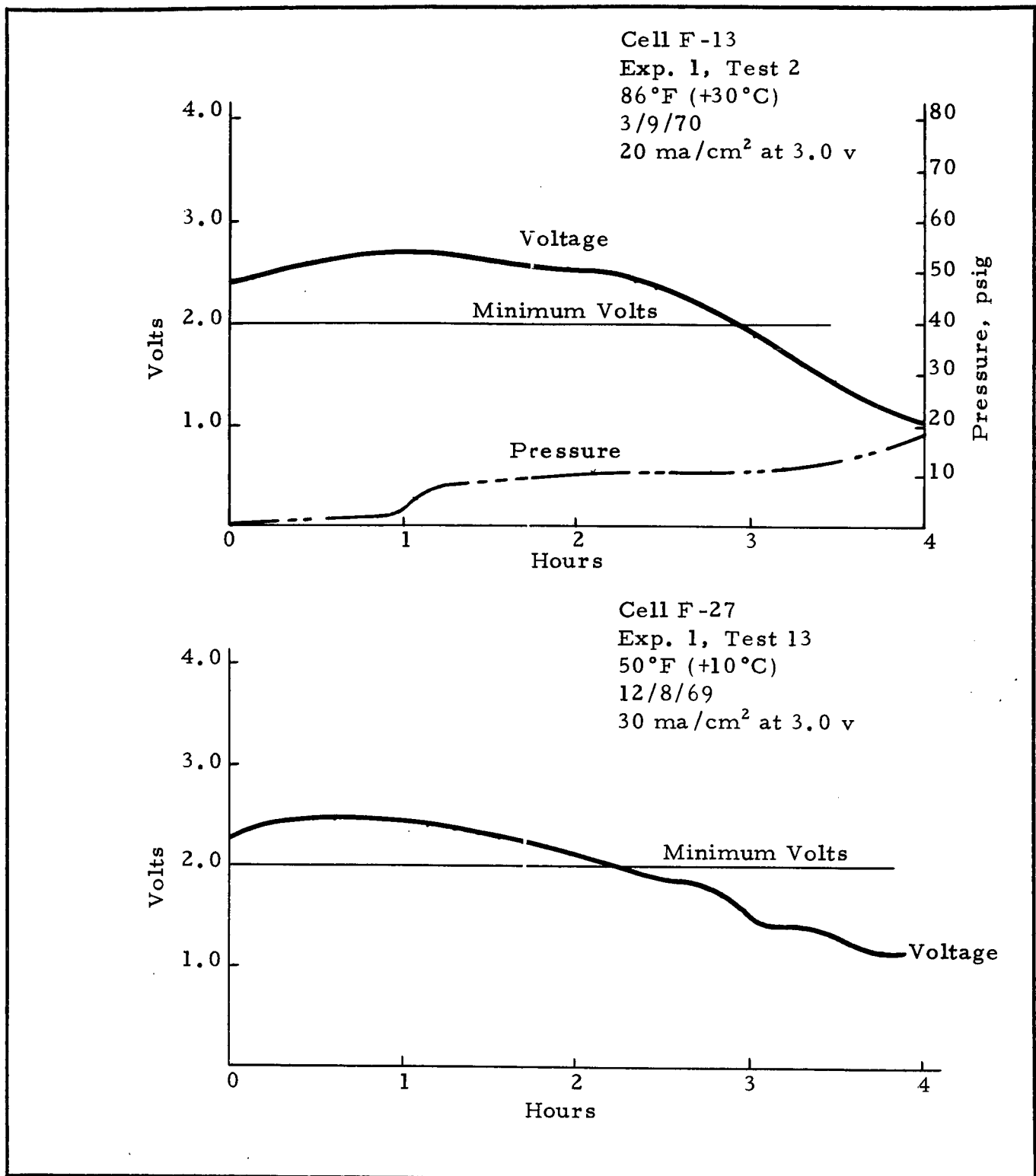


FIGURE 7

Glass Tube Cell Test Fixture and Components

This page is reproduced again at the back of this report by a different reproduction method so as to furnish the best possible detail to the user.

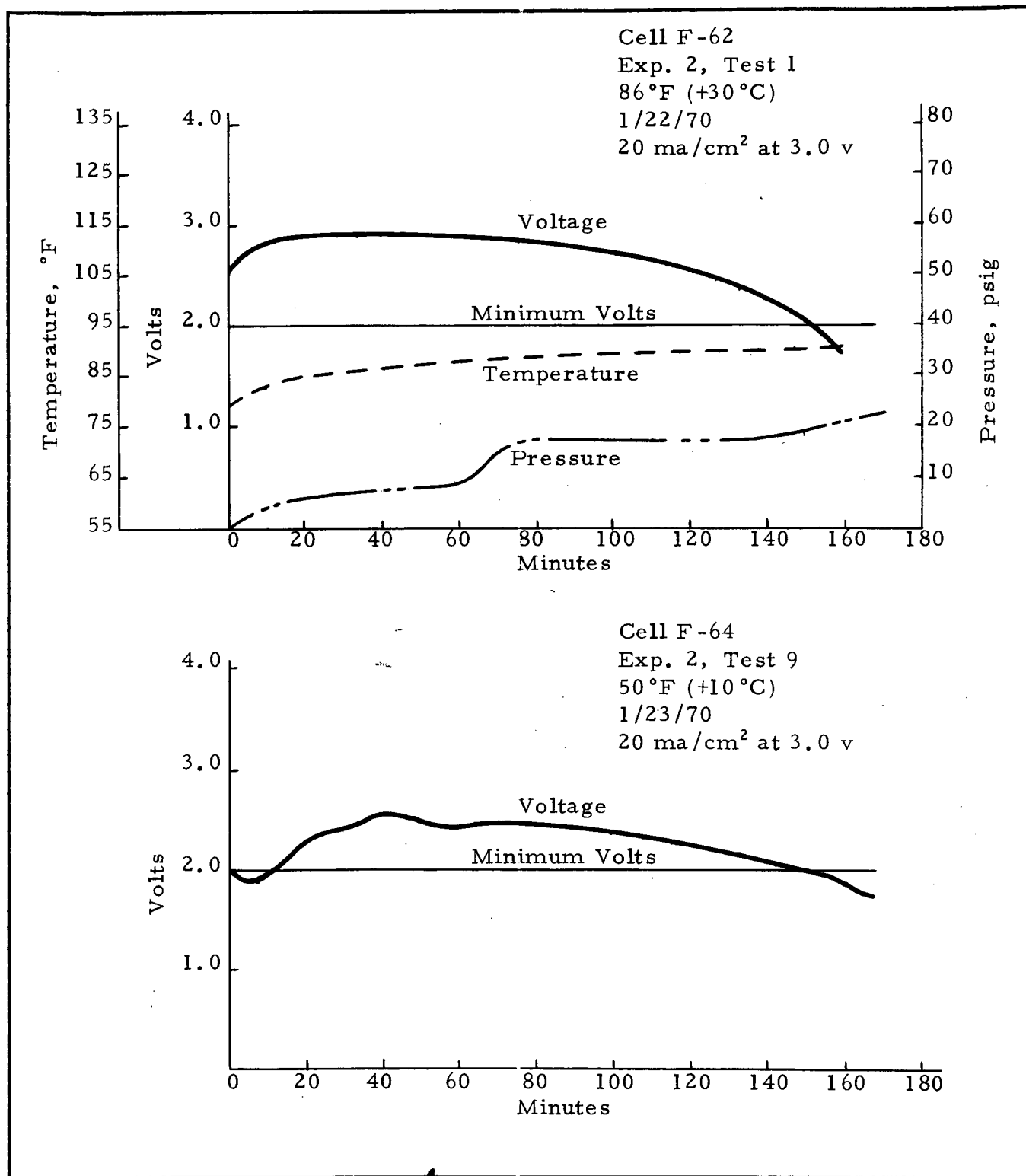


FORM FM-100

FIGURE 8

Discharge Performance of Cells in Experiment #1

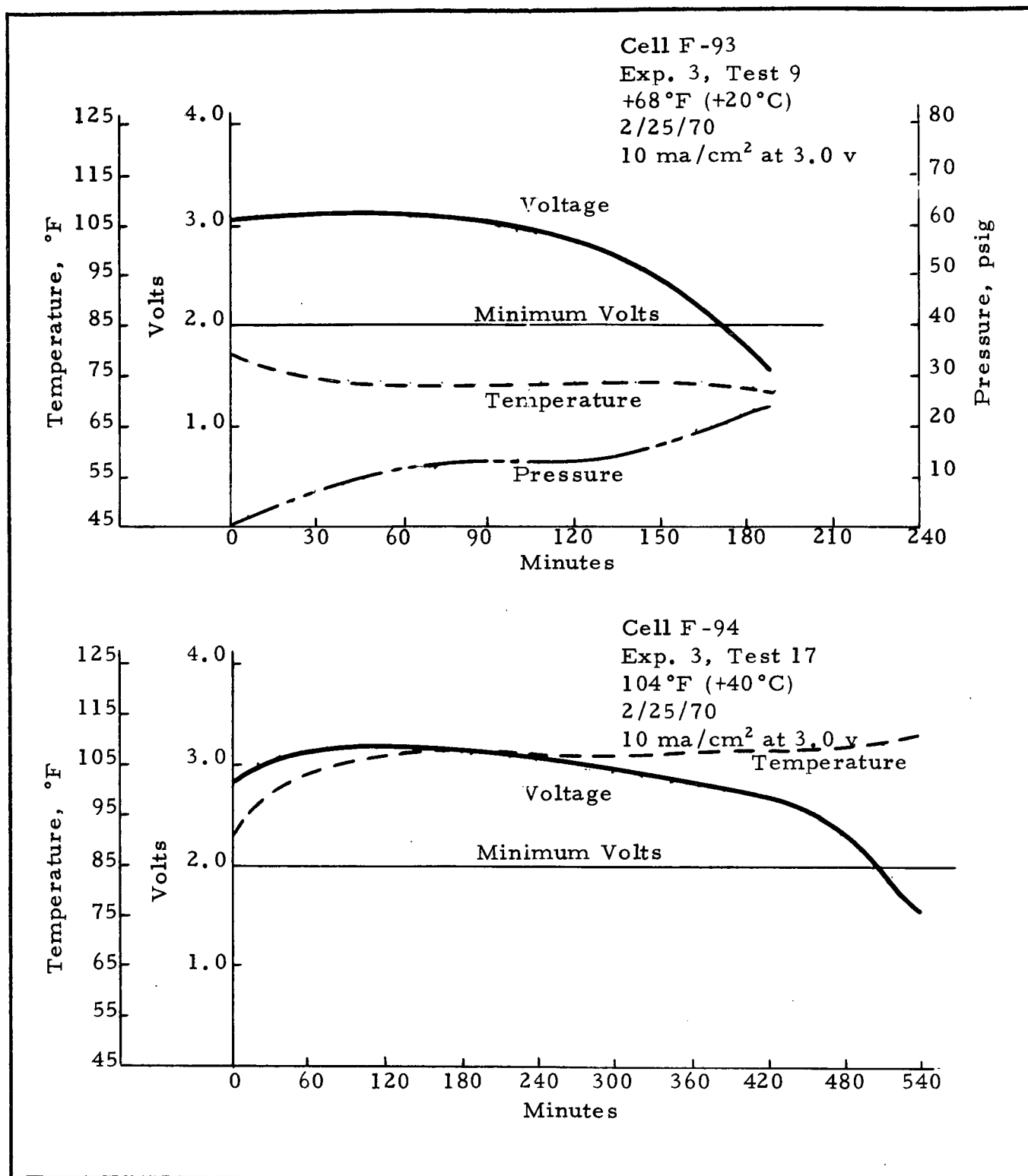




FORM FM-100

FIGURE 9

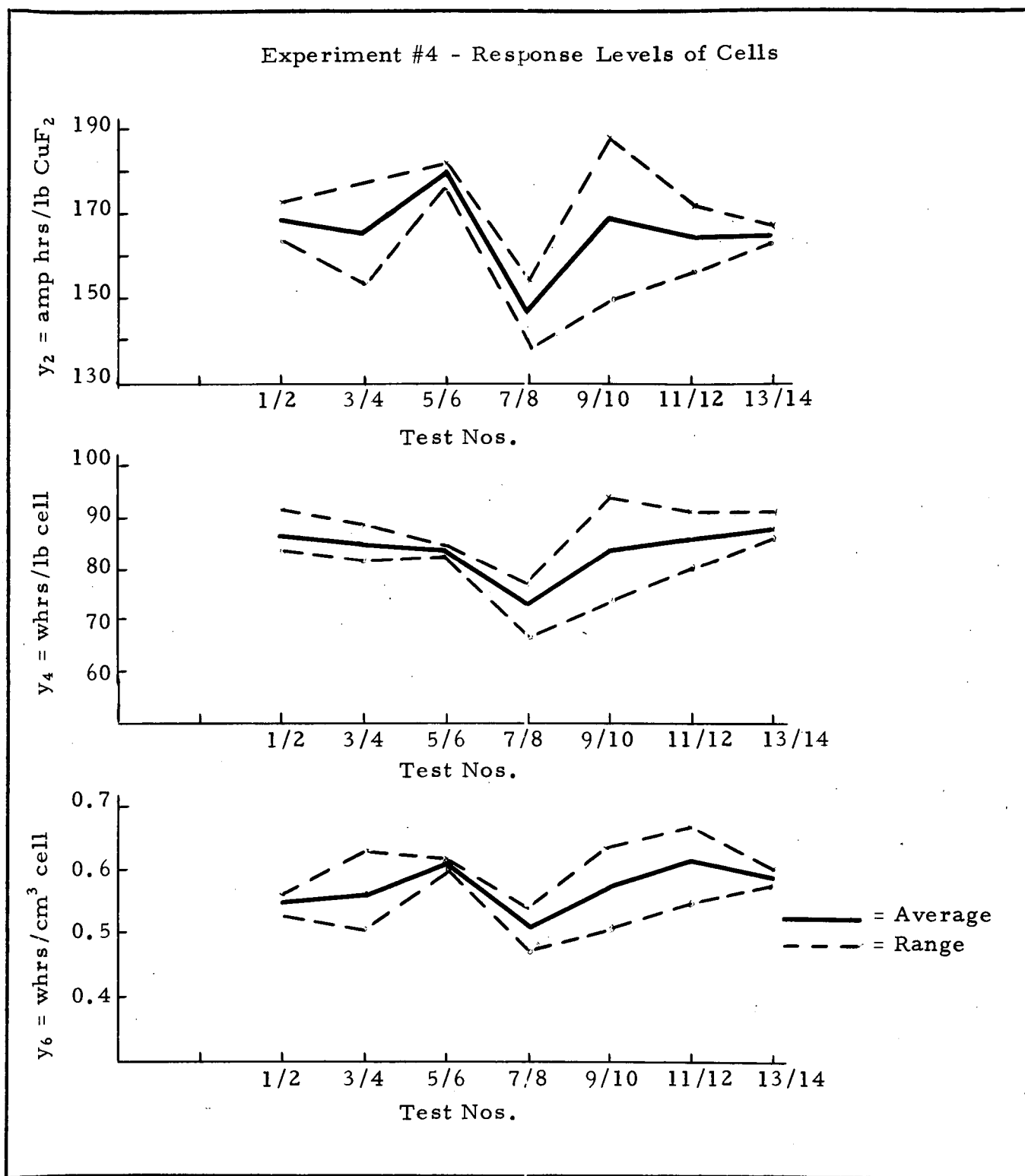
Discharge Performance of Cells in Experiment #2



FORM FM-100

FIGURE 10

Discharge Performance of Cells in Experiment #3



FORM FM-100

FIGURE 11

Response Levels of Cells in Experiment #4

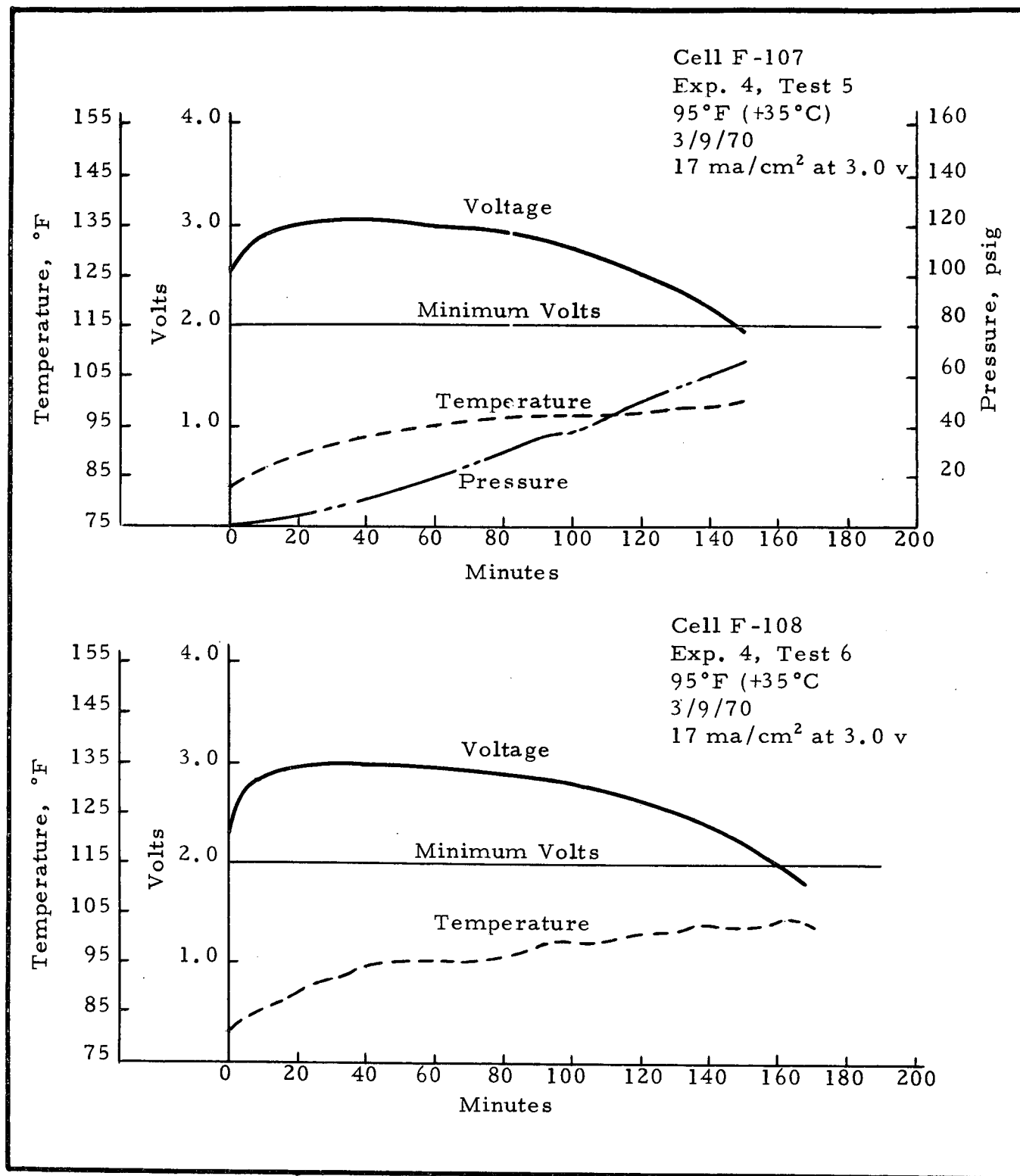
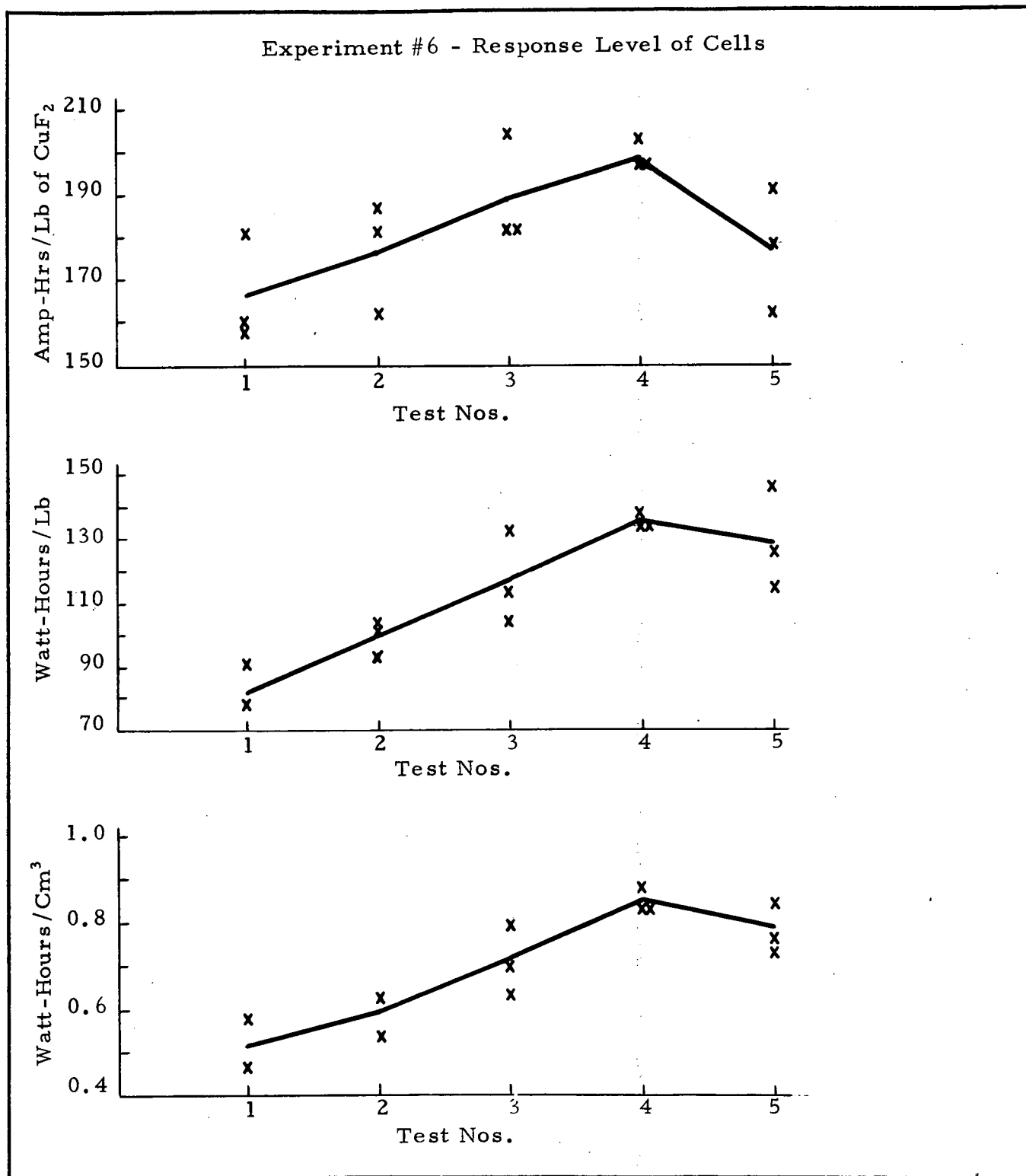


FIGURE 12

Discharge Performance of Cells in Experiment #4



FORM FM-100

FIGURE 13

Response Levels of Cells in Experiment #6

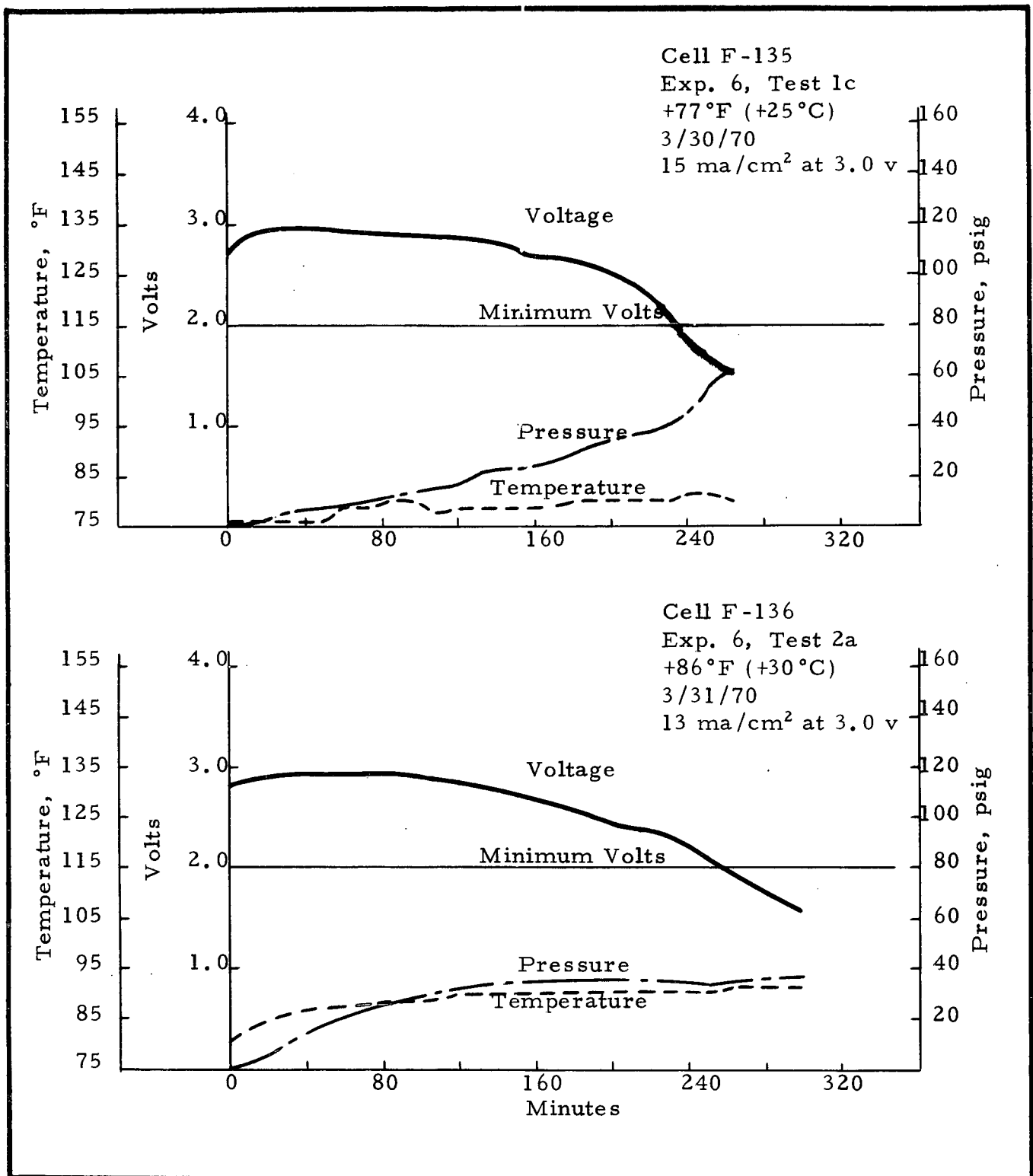


FIGURE 14

Discharge Performance of Cells in Experiment #6

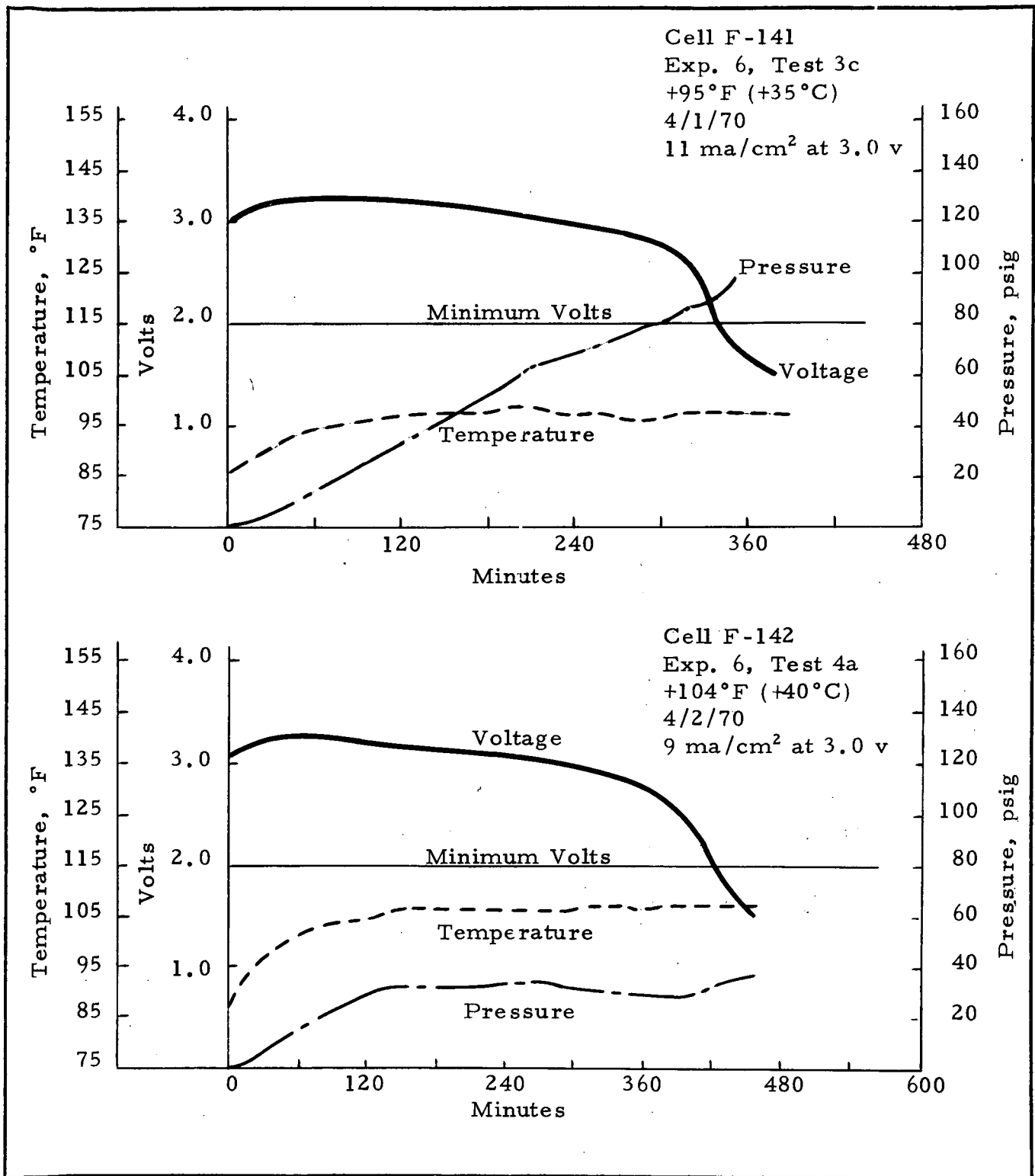
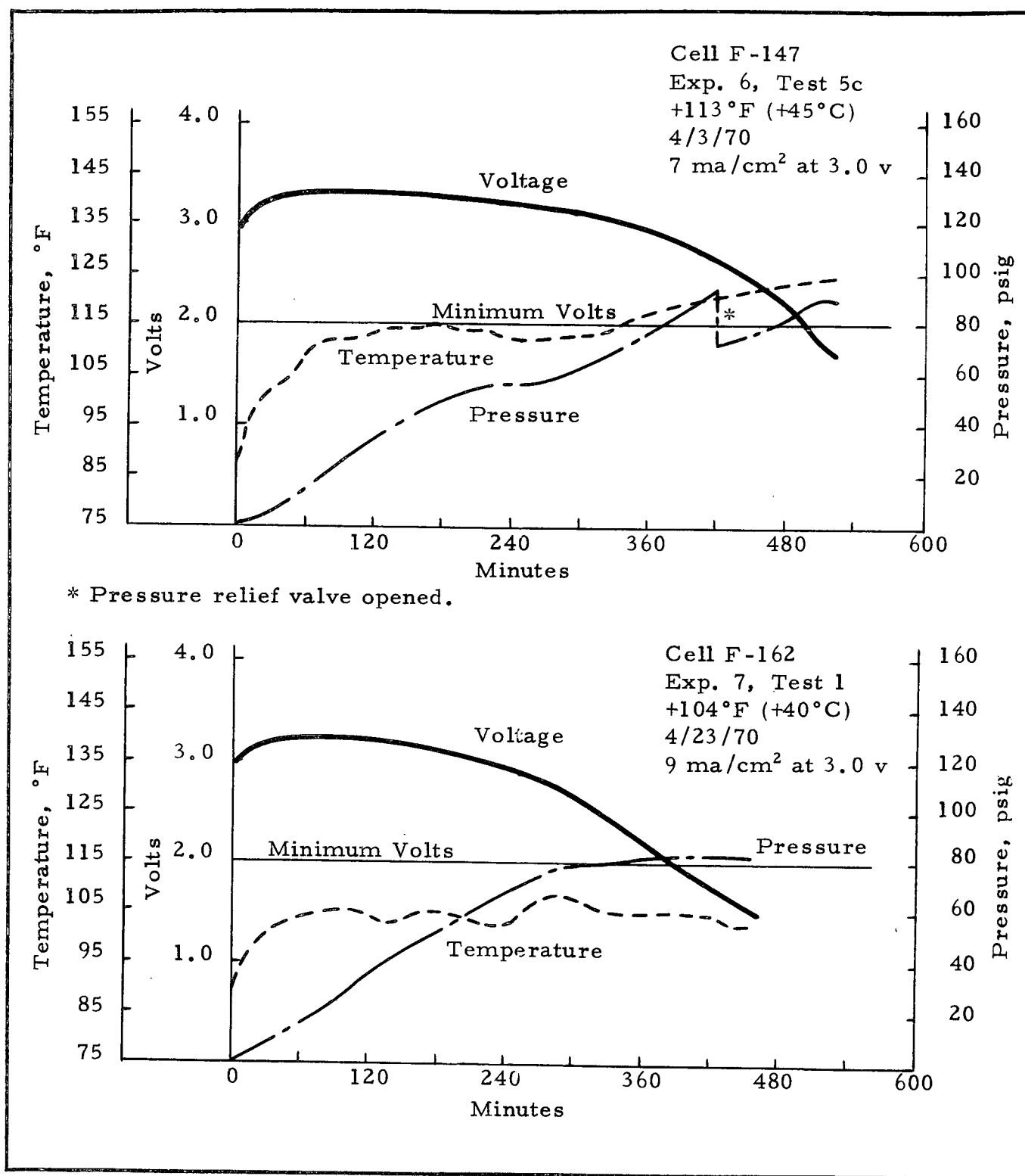


FIGURE 15

Discharge Performance of Cells in Experiment #6



FORM FM-100

FIGURE 16

Discharge Performance of Cells in Experiments #6 and #7



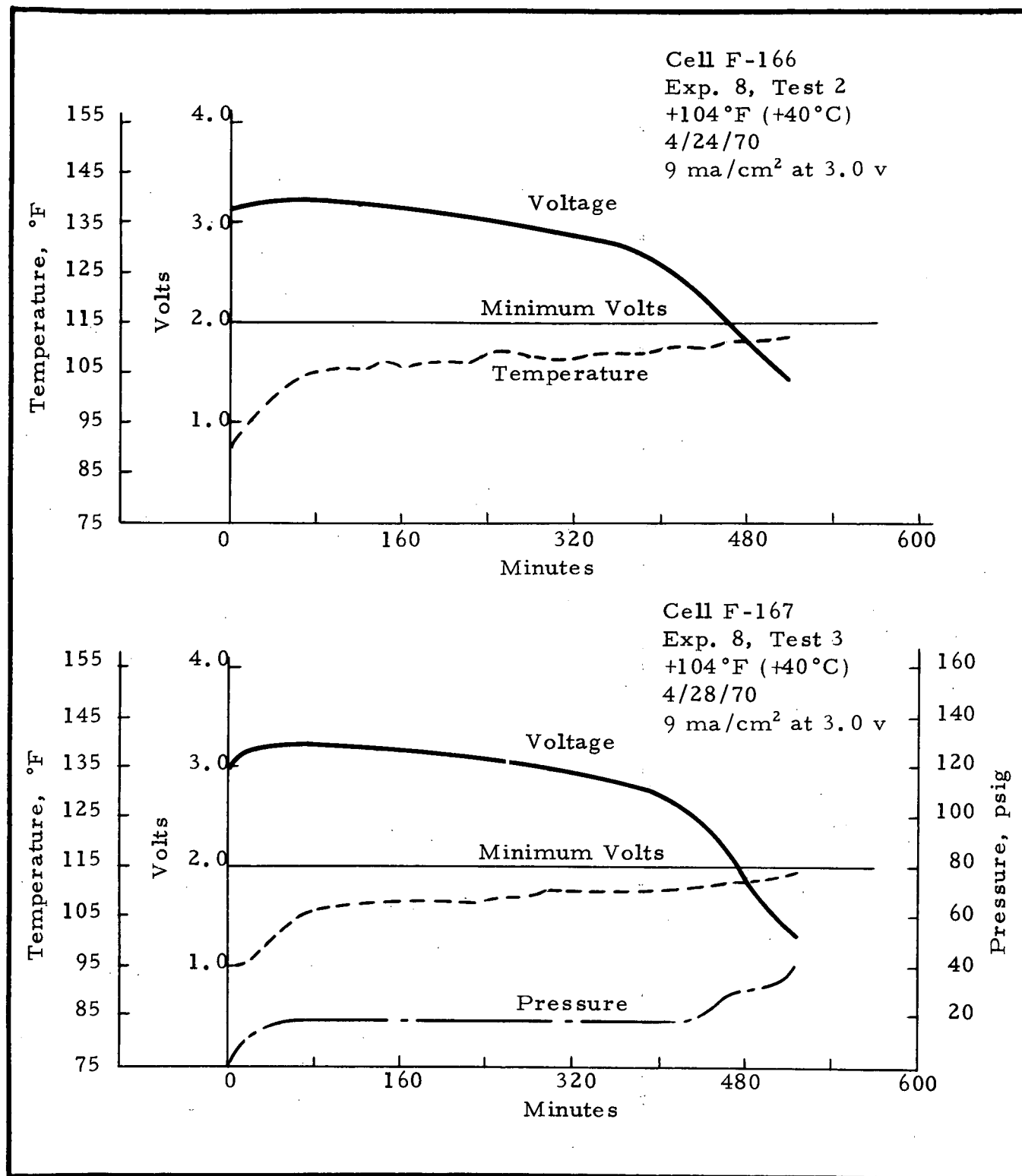
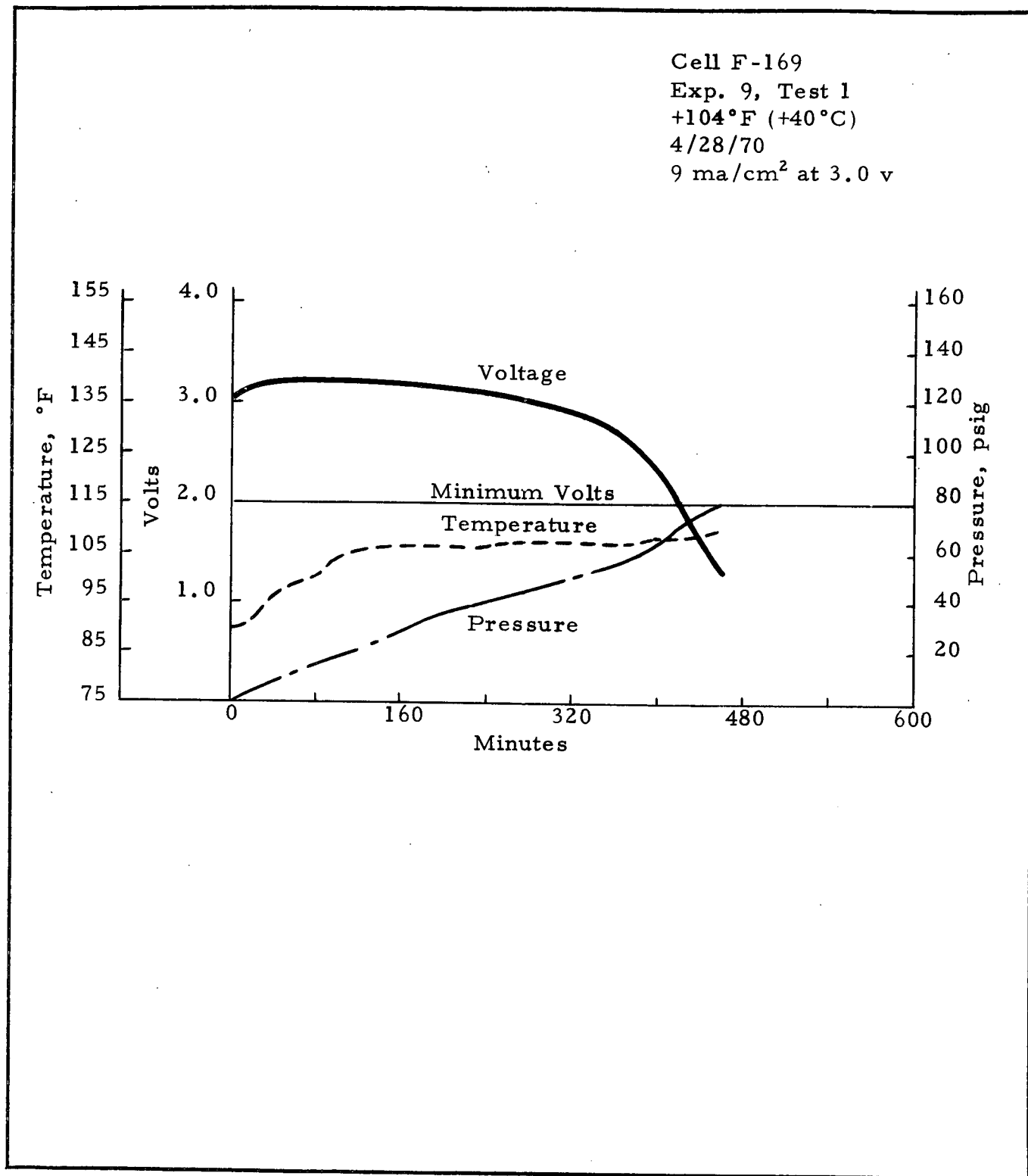


FIGURE 17

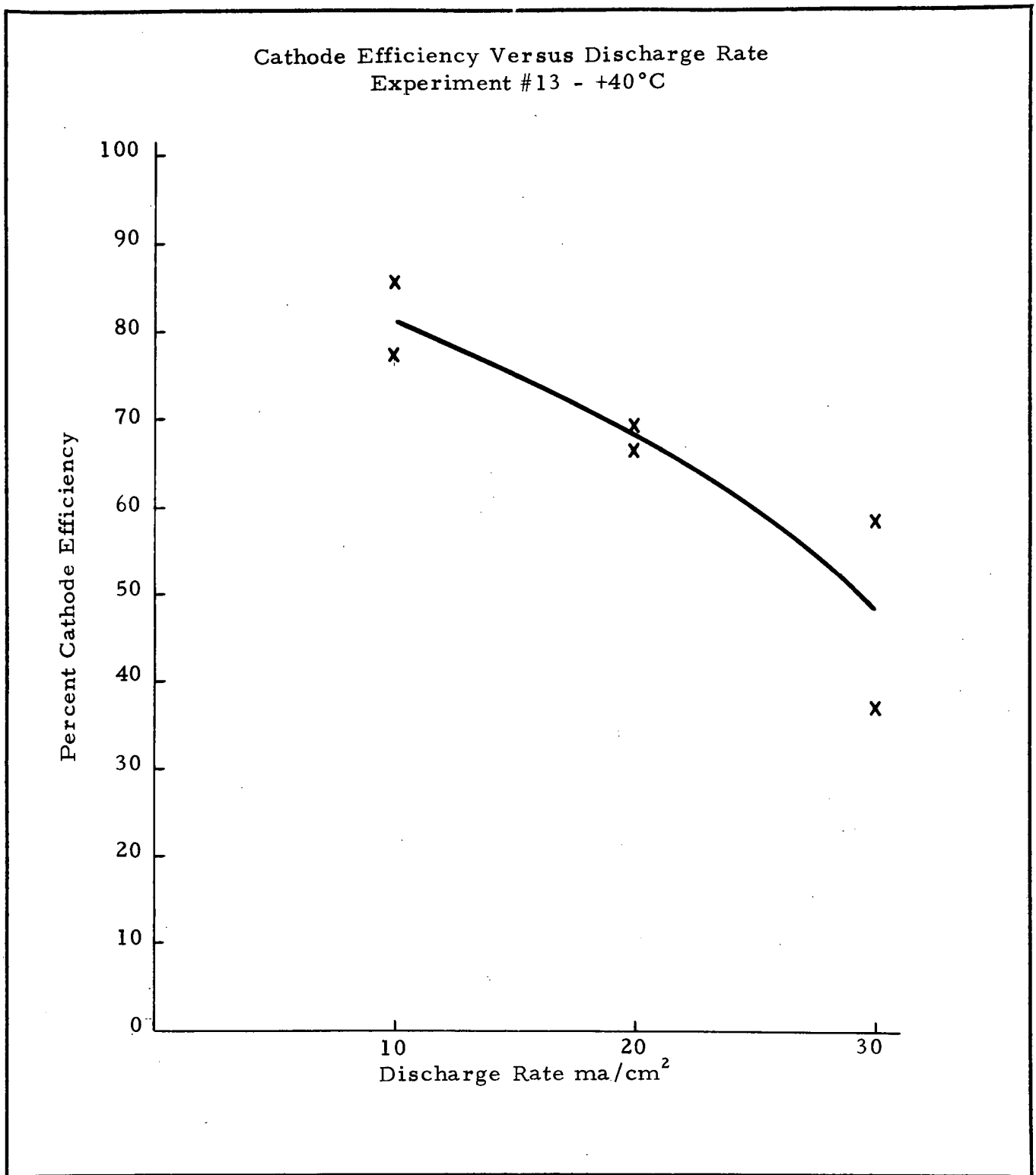
Discharge Performance of Cells in Experiment #8



FORM FM-100

FIGURE 18

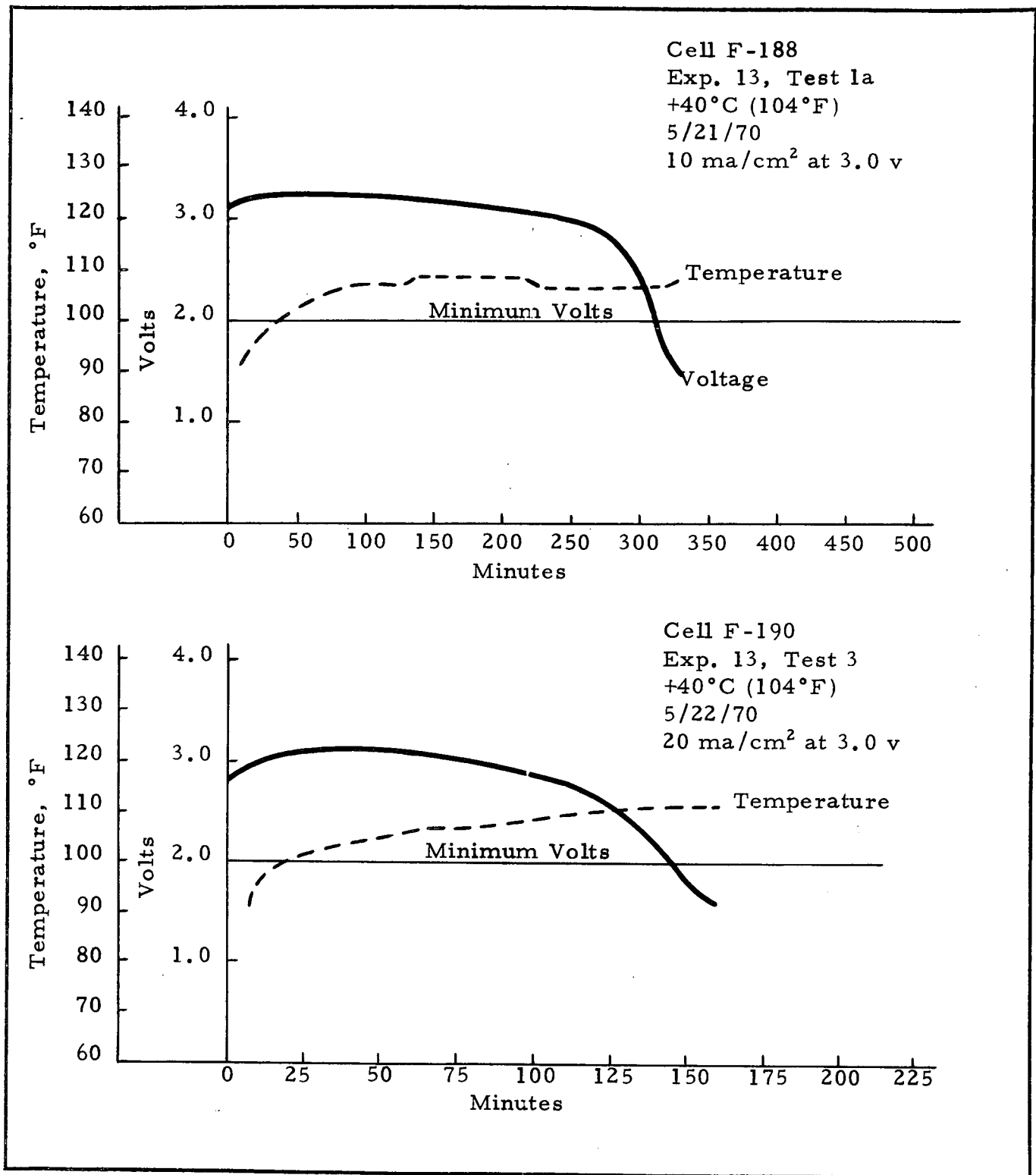
Discharge Performance of Cells in Experiment #9



FORM FM-100

FIGURE 19

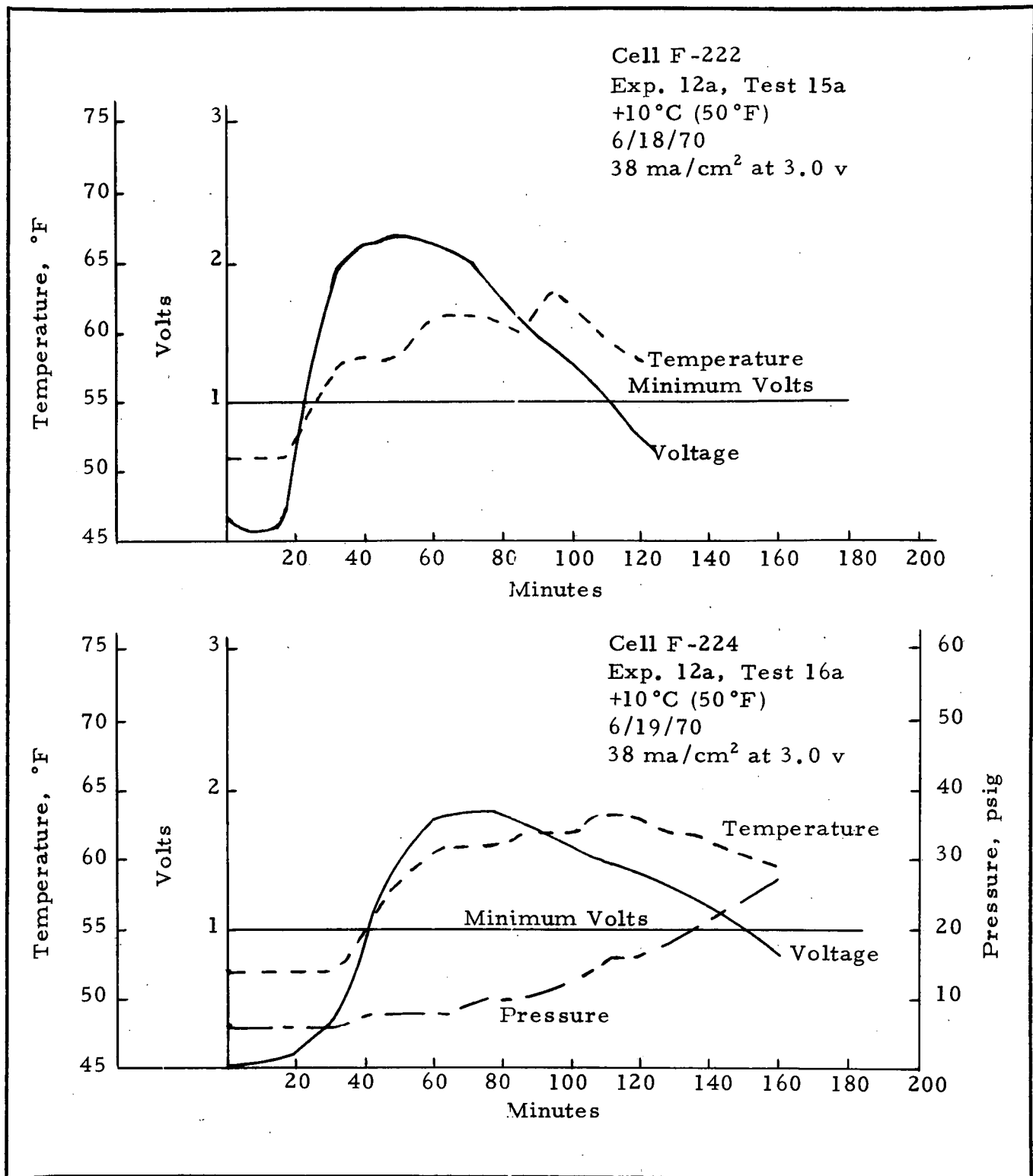
Cathode Efficiency Versus Discharge Rate - Experiment #13



FORM FM-100

FIGURE 20

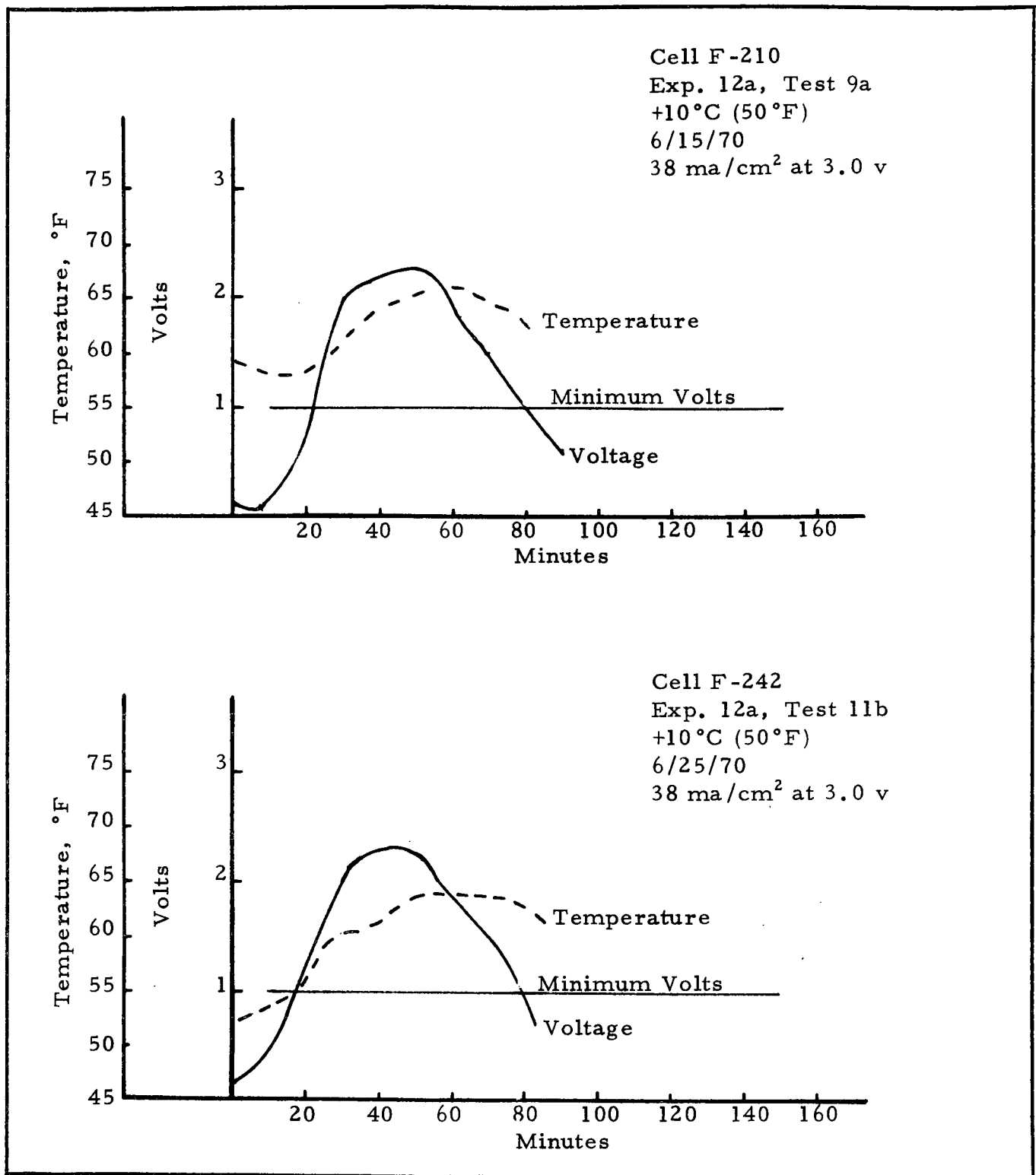
Discharge Performance of Cells in Experiment #13



FORM FM-100

FIGURE 22

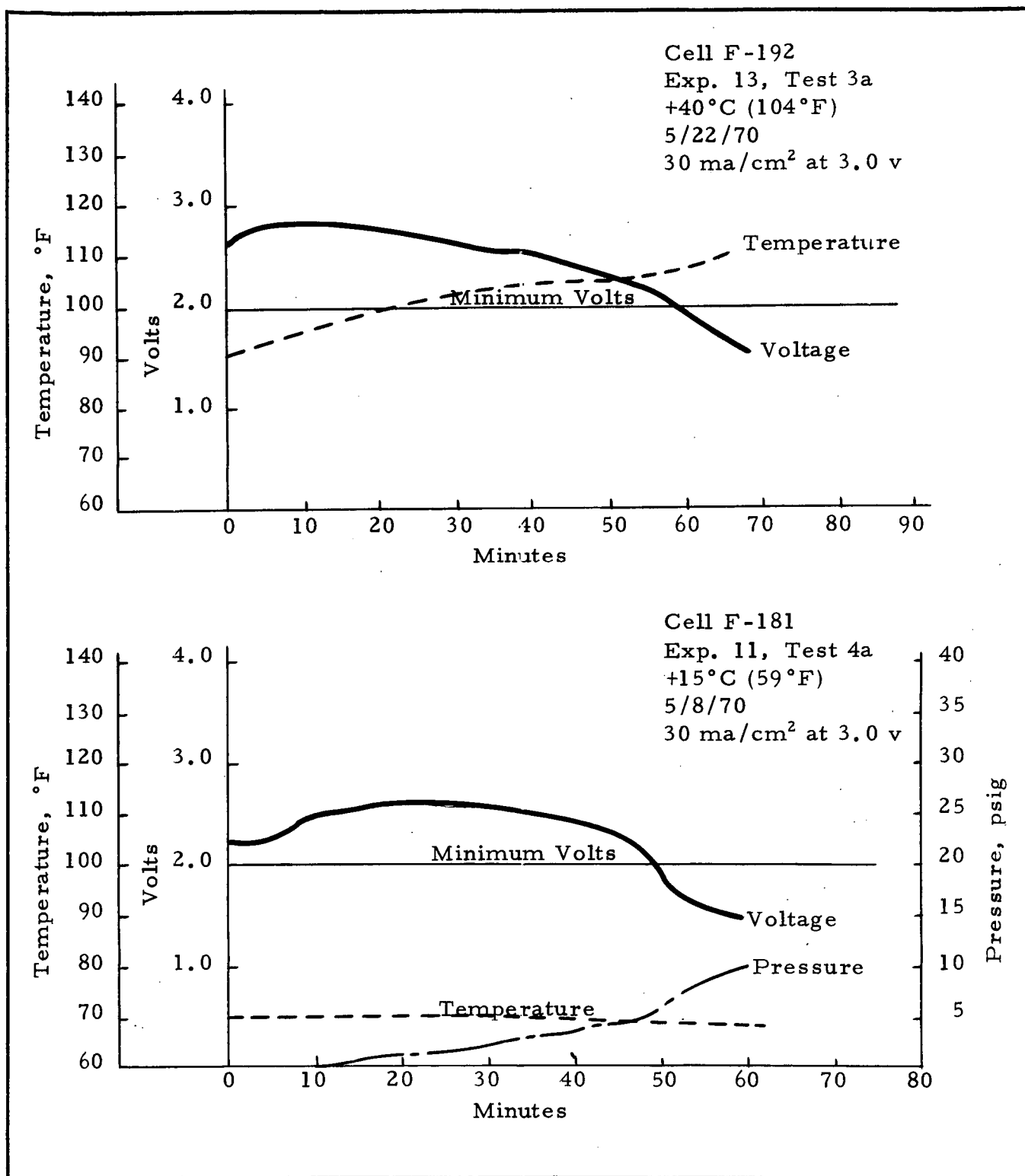
Discharge Performance of Cells in Experiment #12a



FORM FM-100

FIGURE 23

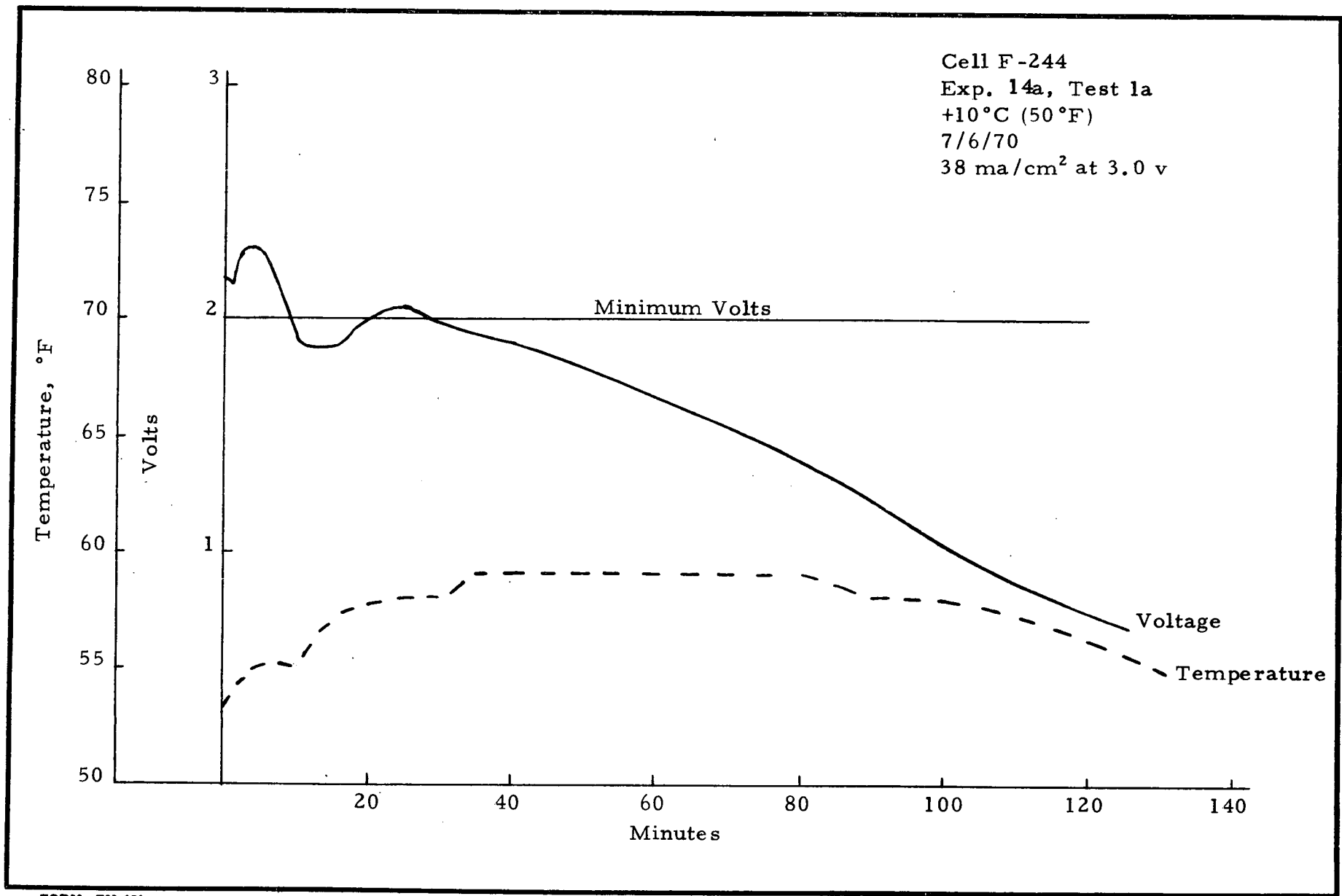
Discharge Performance of Cells in Experiment #12a



FORM FM-100

FIGURE 21

Discharge Performance of Cells in Experiments #11 and #13



FORM FM-101

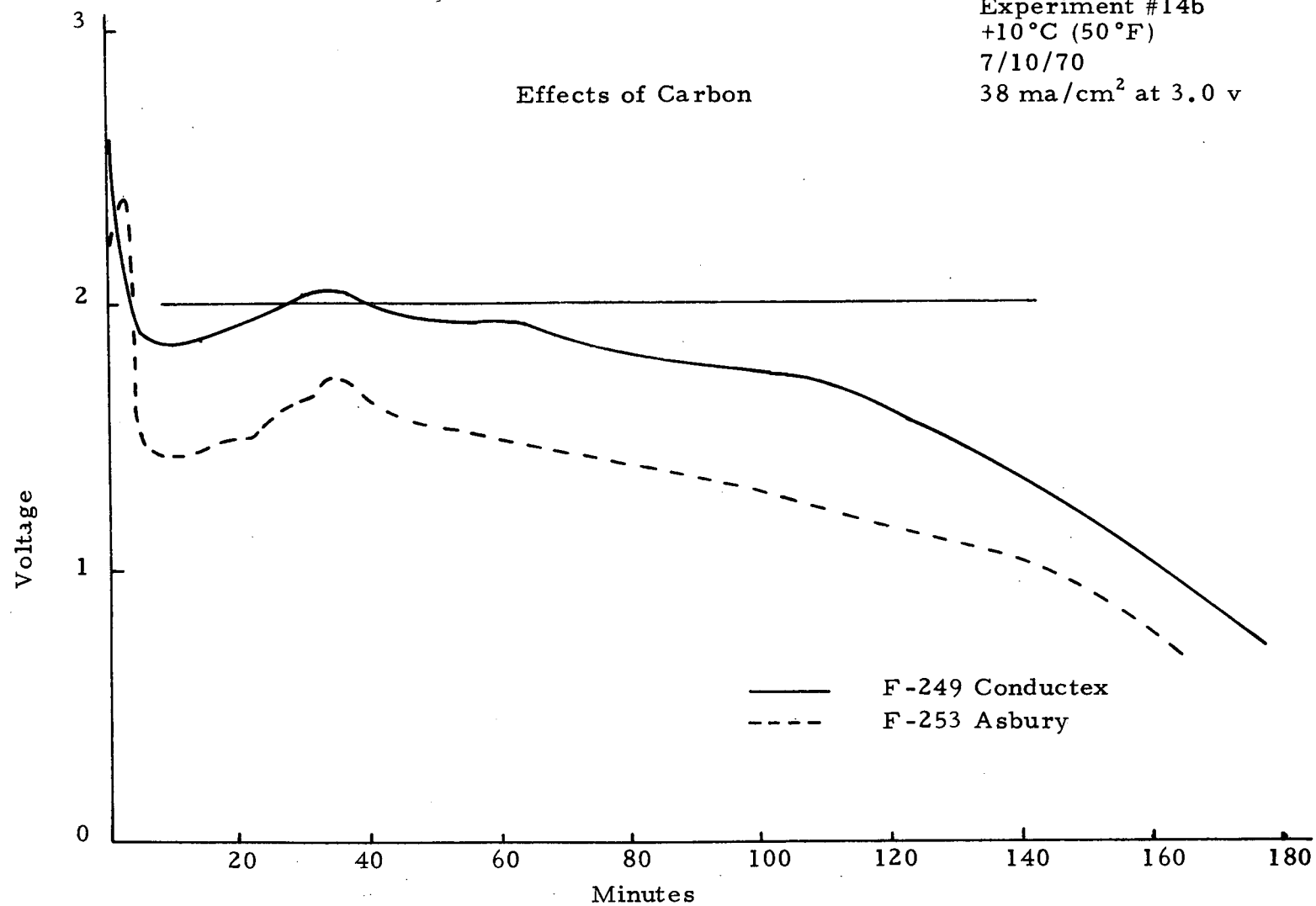
FIGURE 24

Discharge Performance of Cells in Experiment #14a



Effects of Carbon

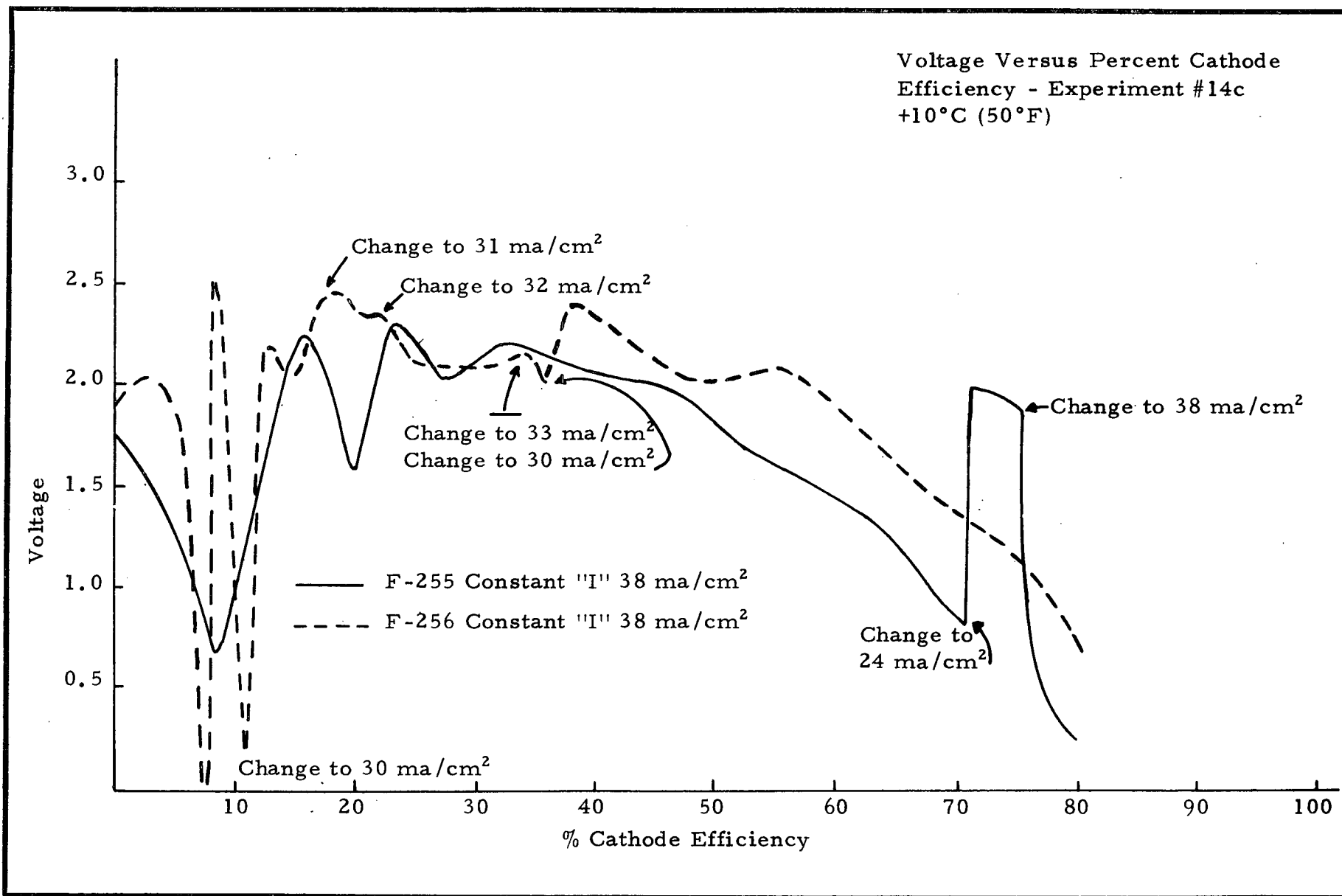
Experiment #14b  
+10°C (50°F)  
7/10/70  
38 ma/cm<sup>2</sup> at 3.0 v



FORM FM-101

FIGURE 25

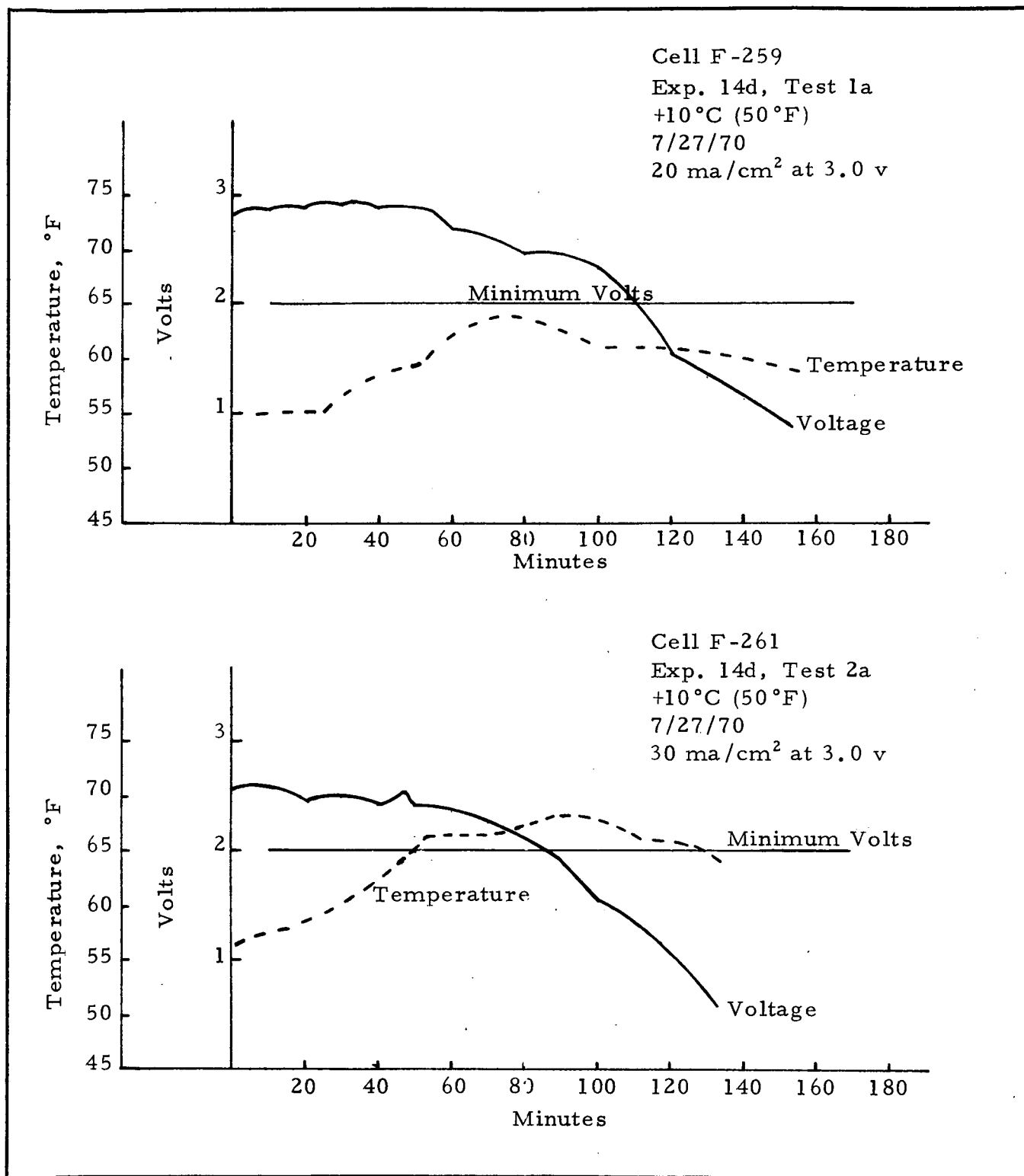
Discharge Performance of Cells in Experiment #14b



FORM FM-101

FIGURE 26

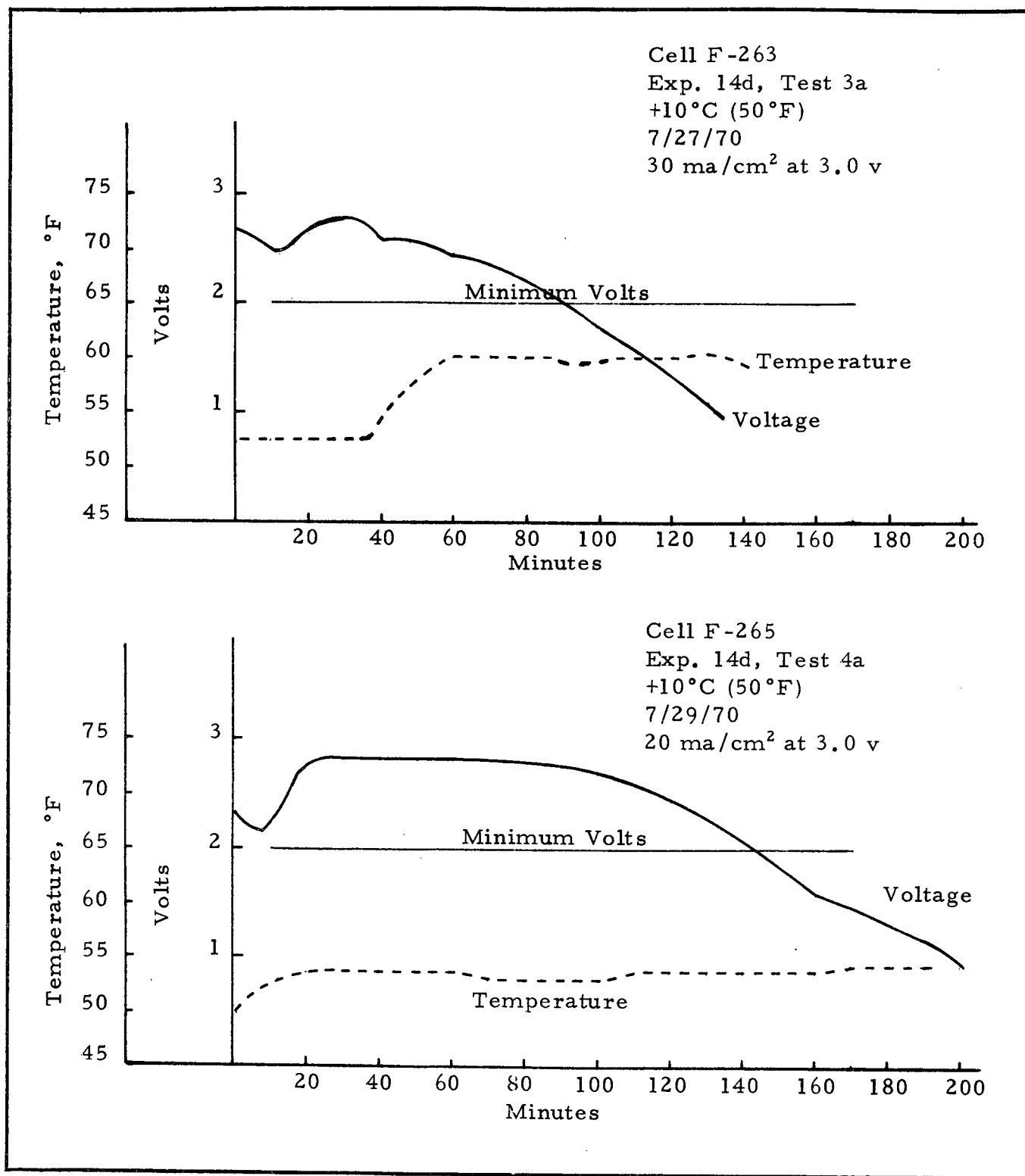
Voltage Versus Percent Cathode Efficiency - Experiment #14c



FORM FM-100

FIGURE 27

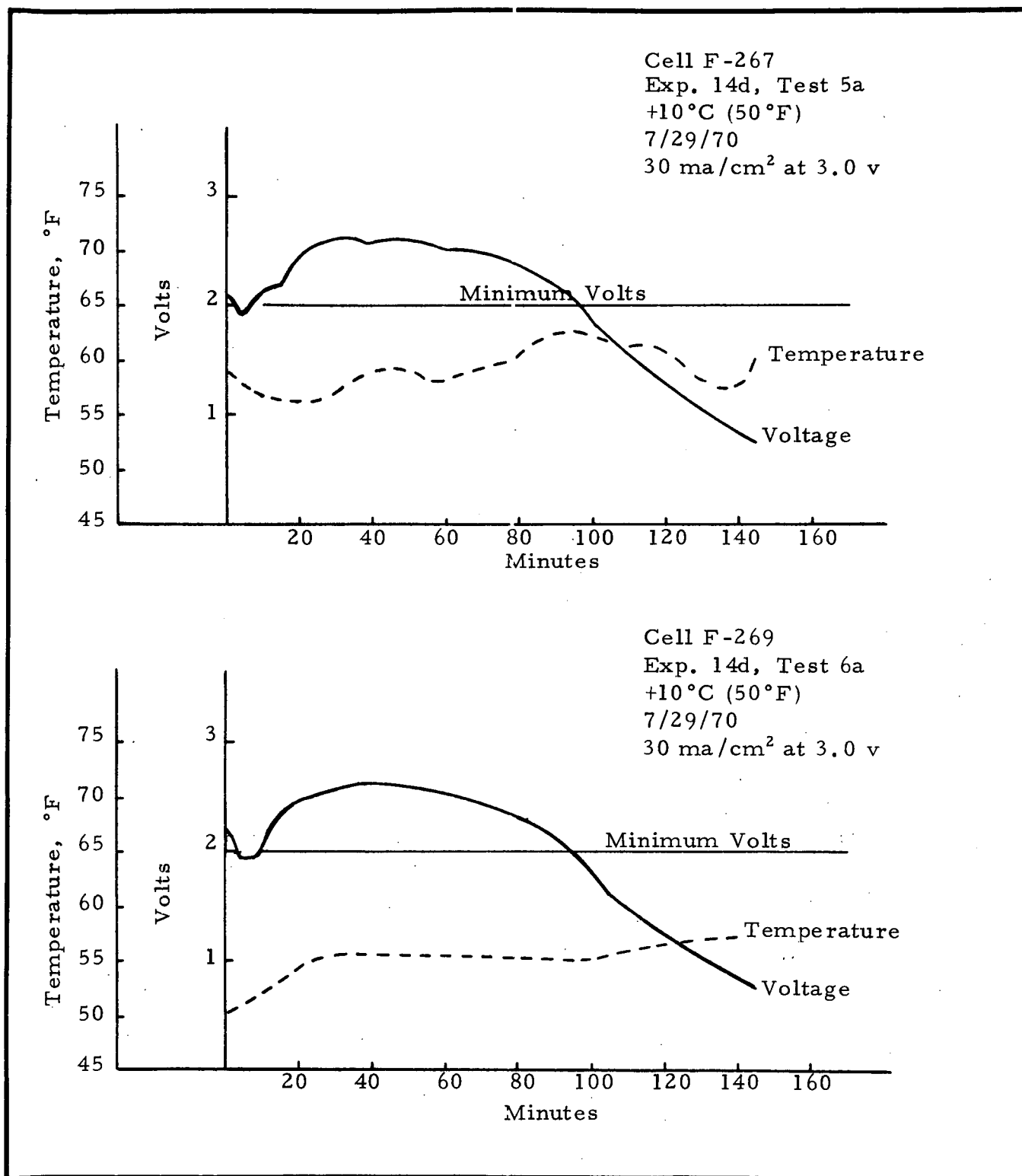
Discharge Performance of Cells in Experiment #14d



FORM FM-100

FIGURE 28

Discharge Performance of Cells in Experiment #14d



FORM FM-100

FIGURE 29

Discharge Performance of Cells in Experiment #14d

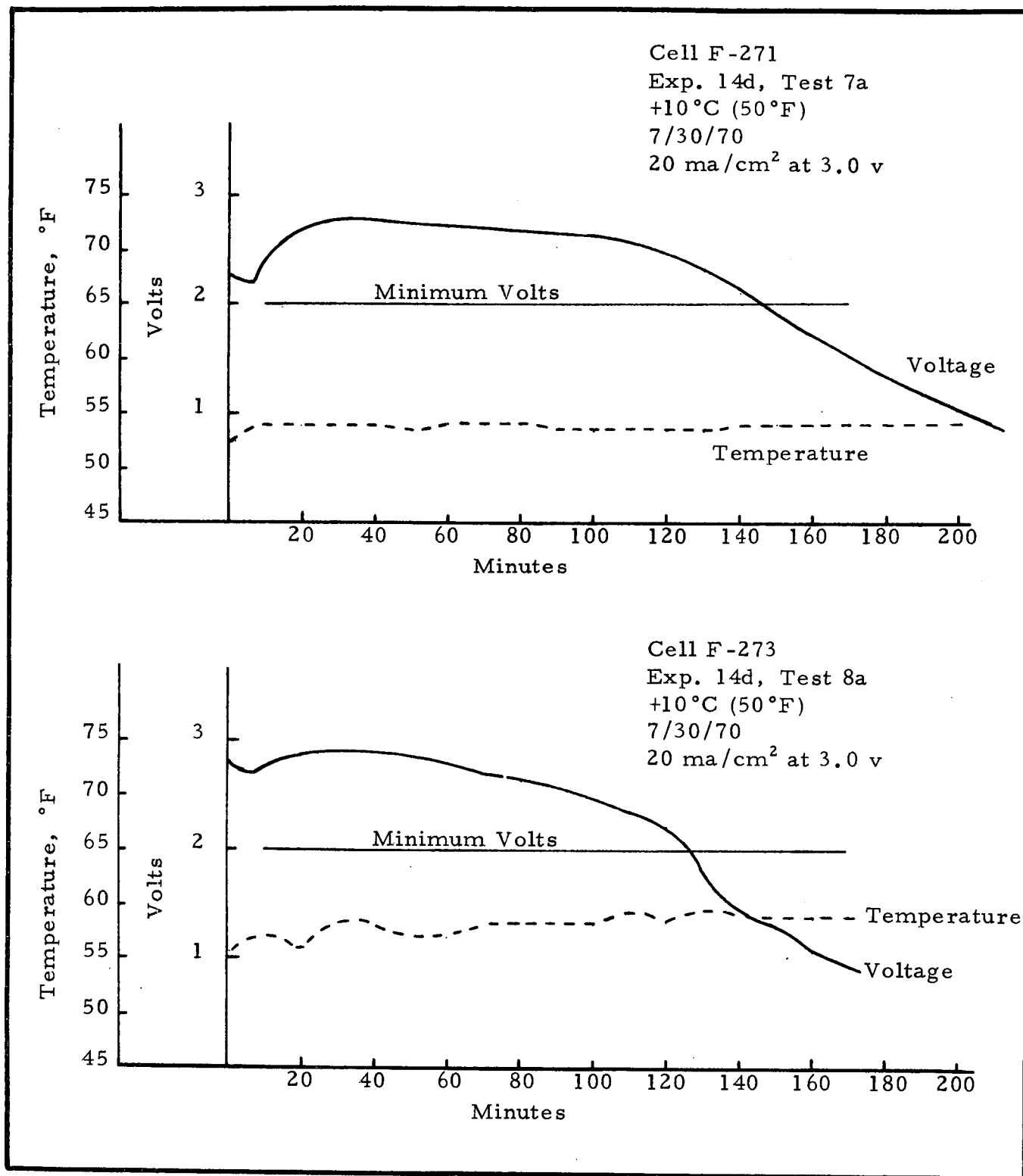
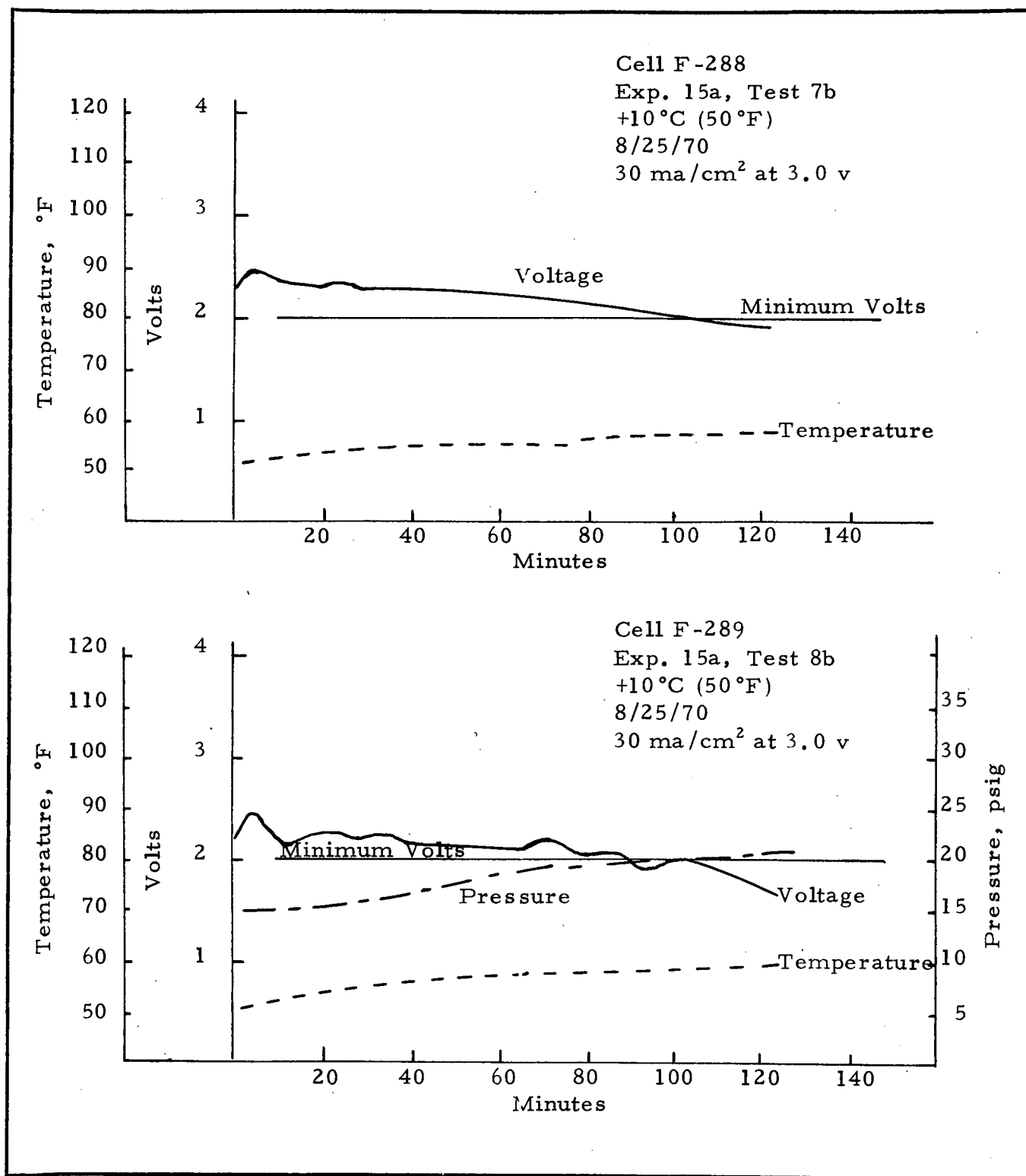


FIGURE 30

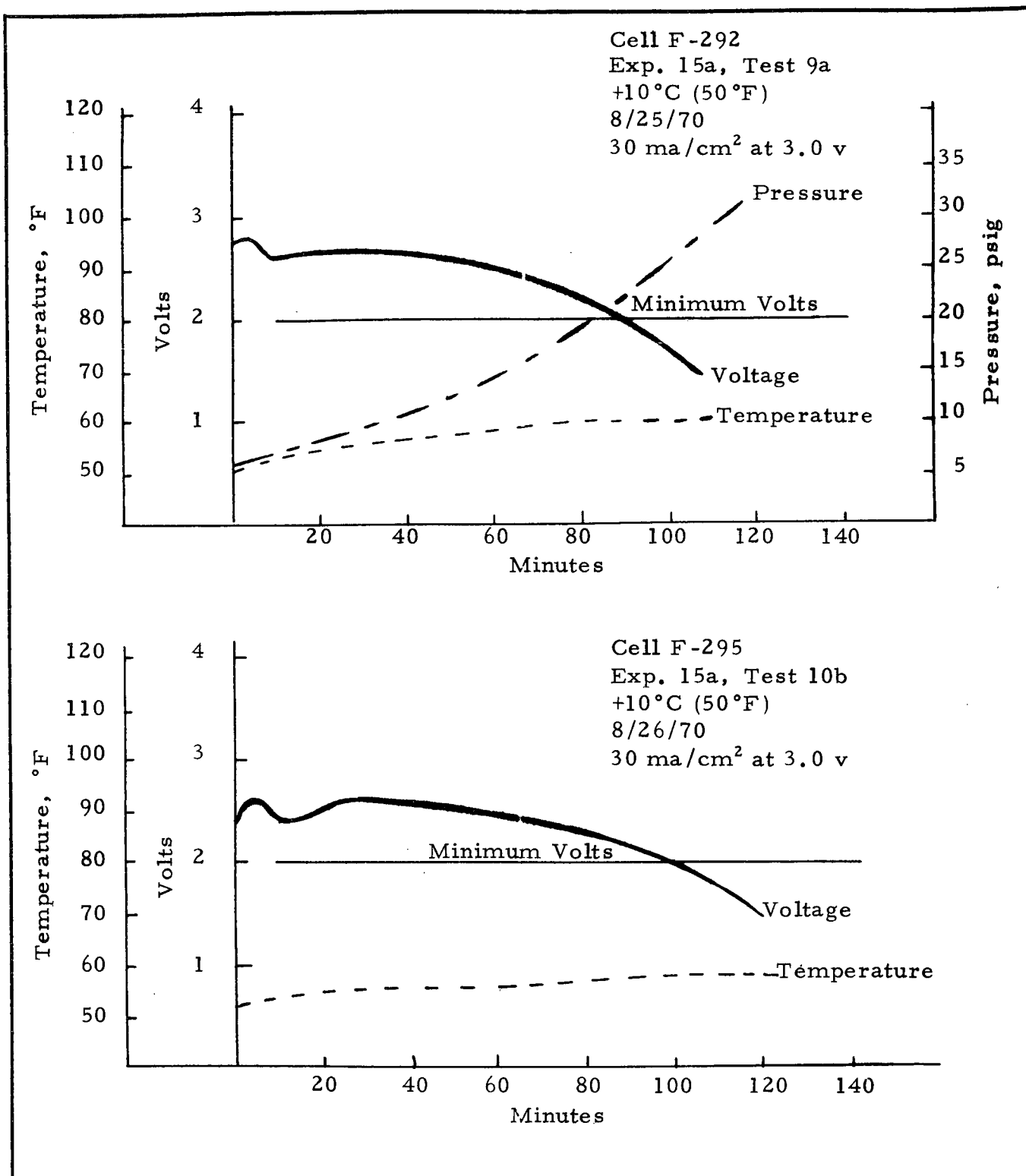
Discharge Performance of Cells in Experiment #14d



FORM FM-100

FIGURE 31

Discharge Performance of Cells in Experiment #15a

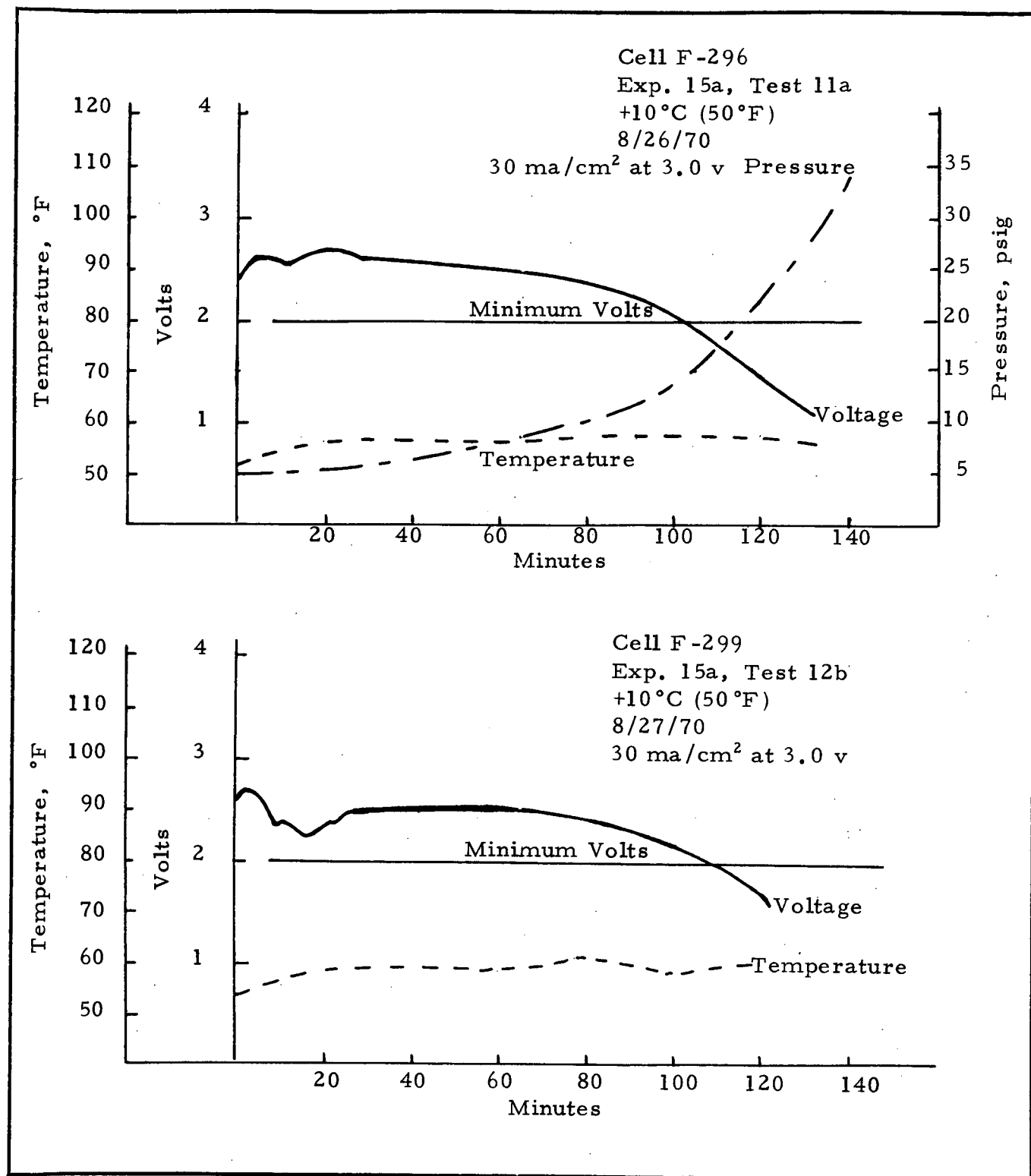


FORM FM-100

FIGURE 32

Discharge Performance of Cells in Experiment #15a

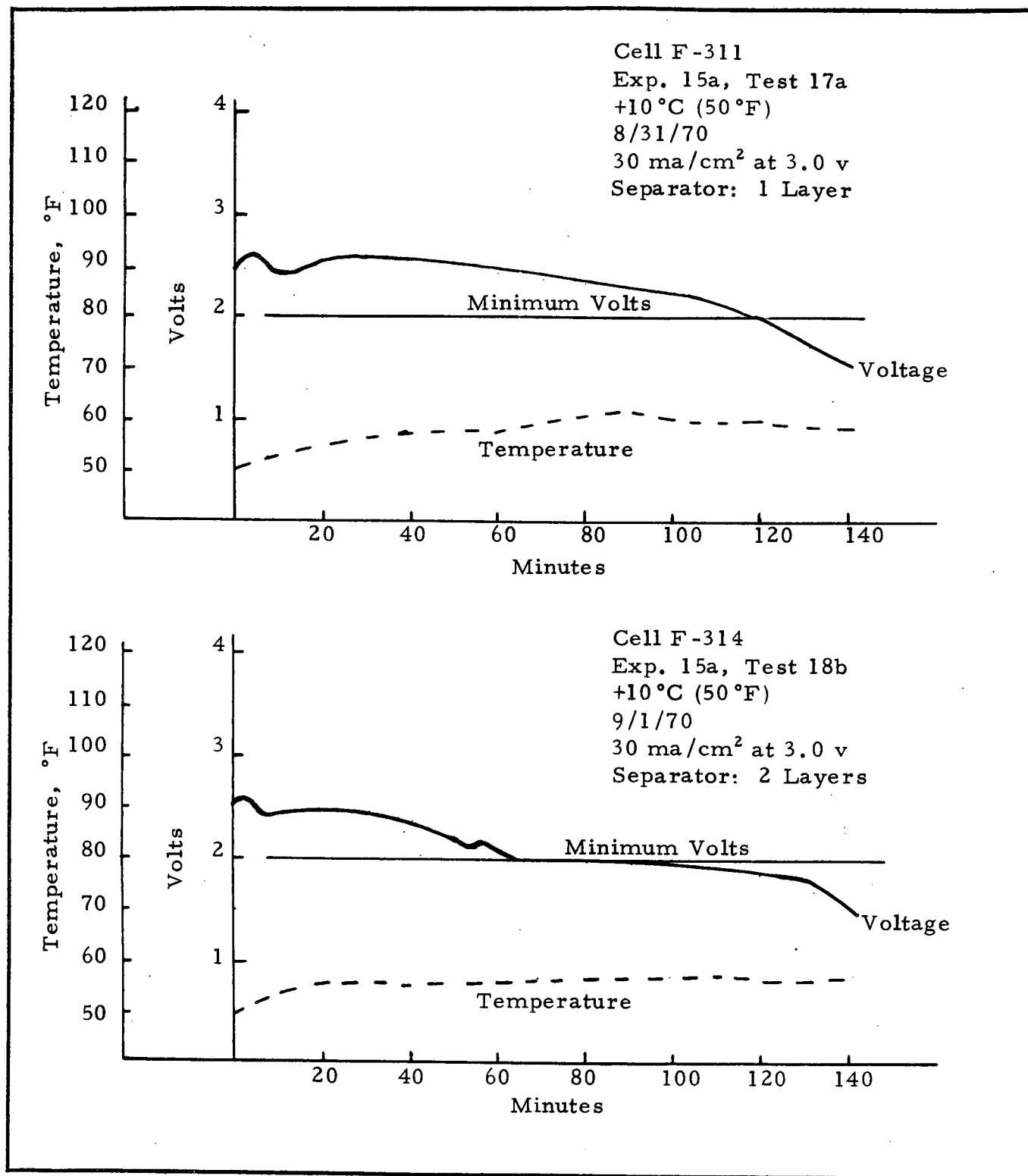




FORM FM-100

FIGURE 33

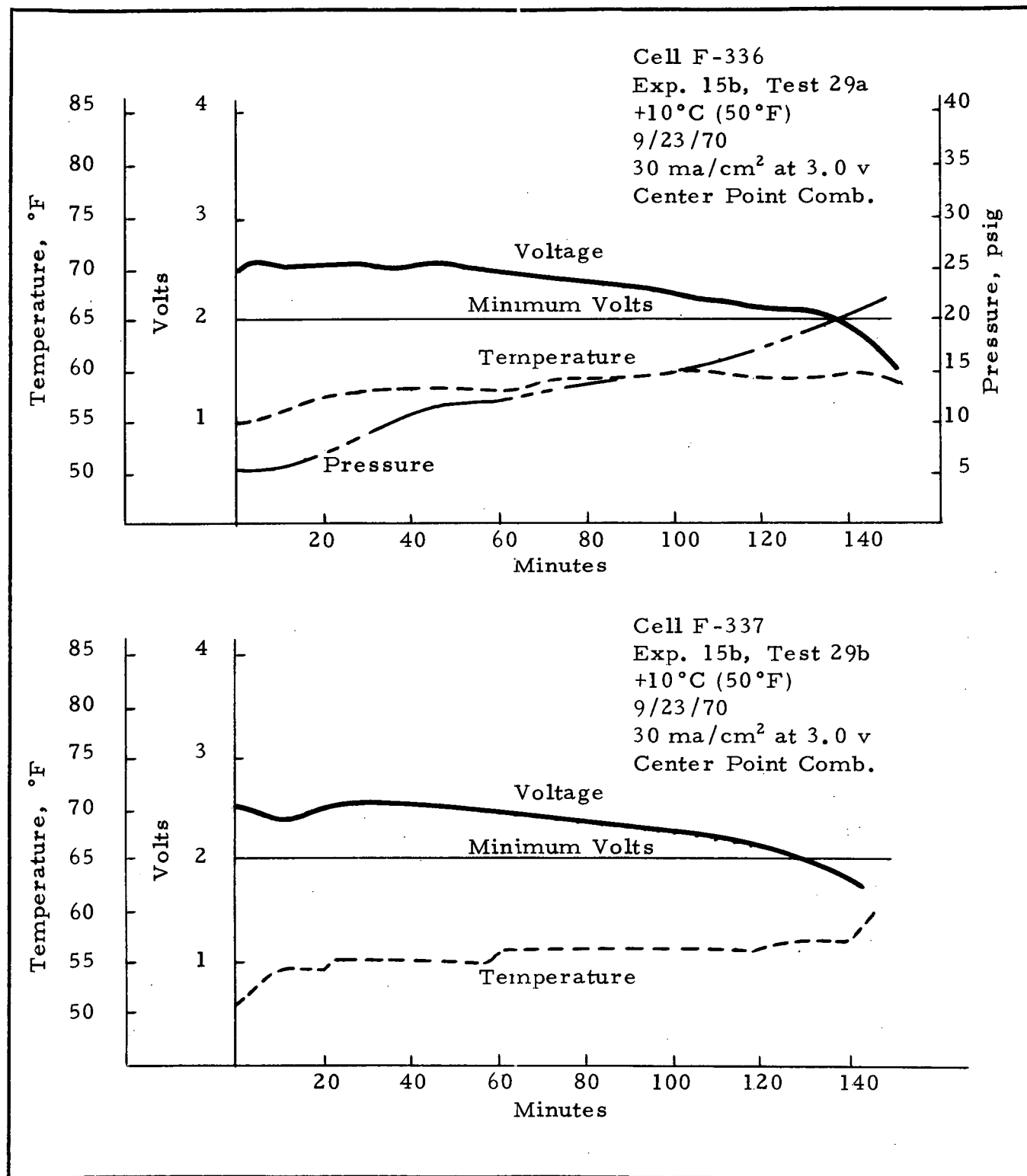
Discharge Performance of Cells in Experiment #15a



FORM FM-100

FIGURE 34

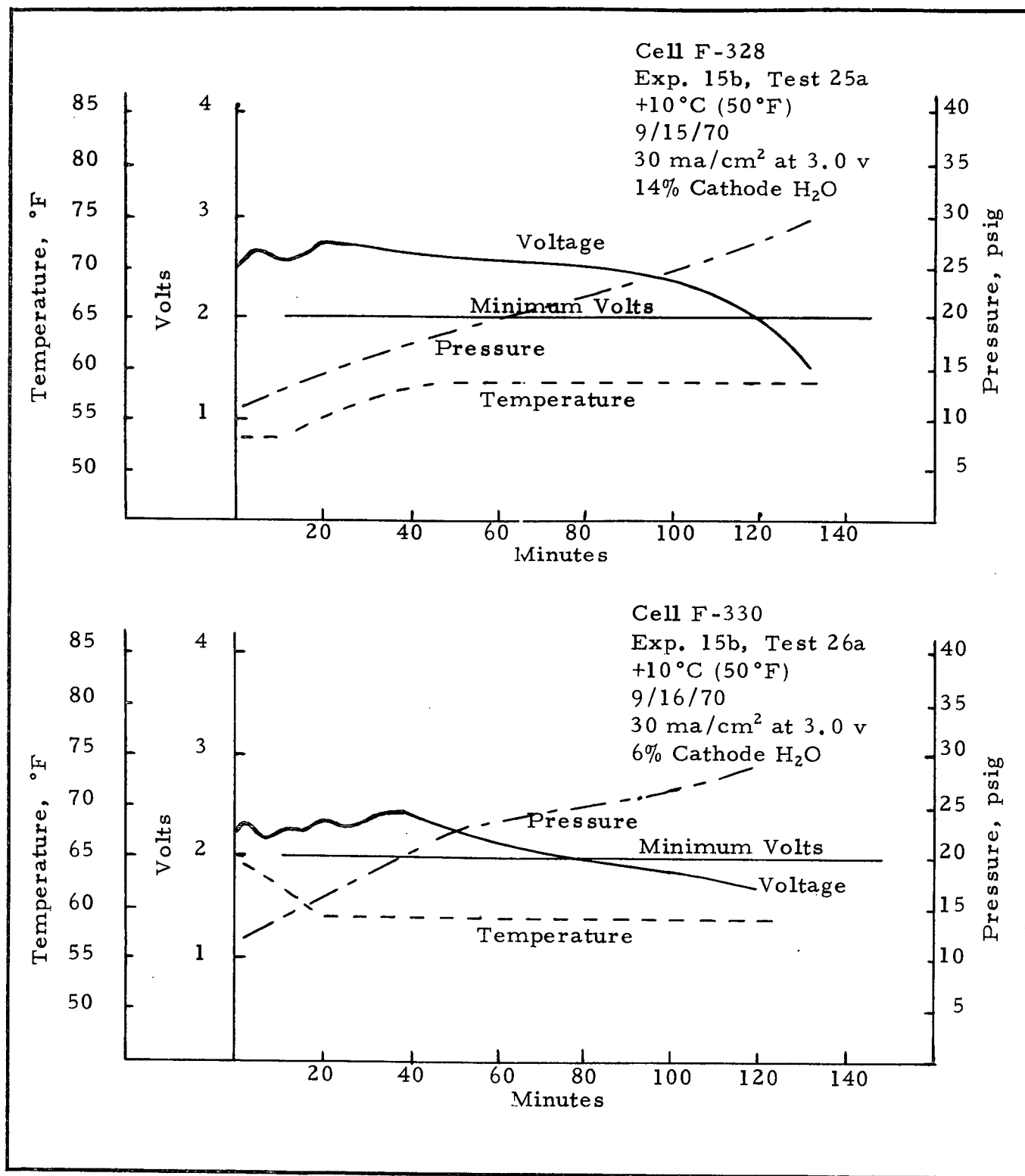
Discharge Performance of Cells in Experiment #15a



FORM FM-100

FIGURE 35

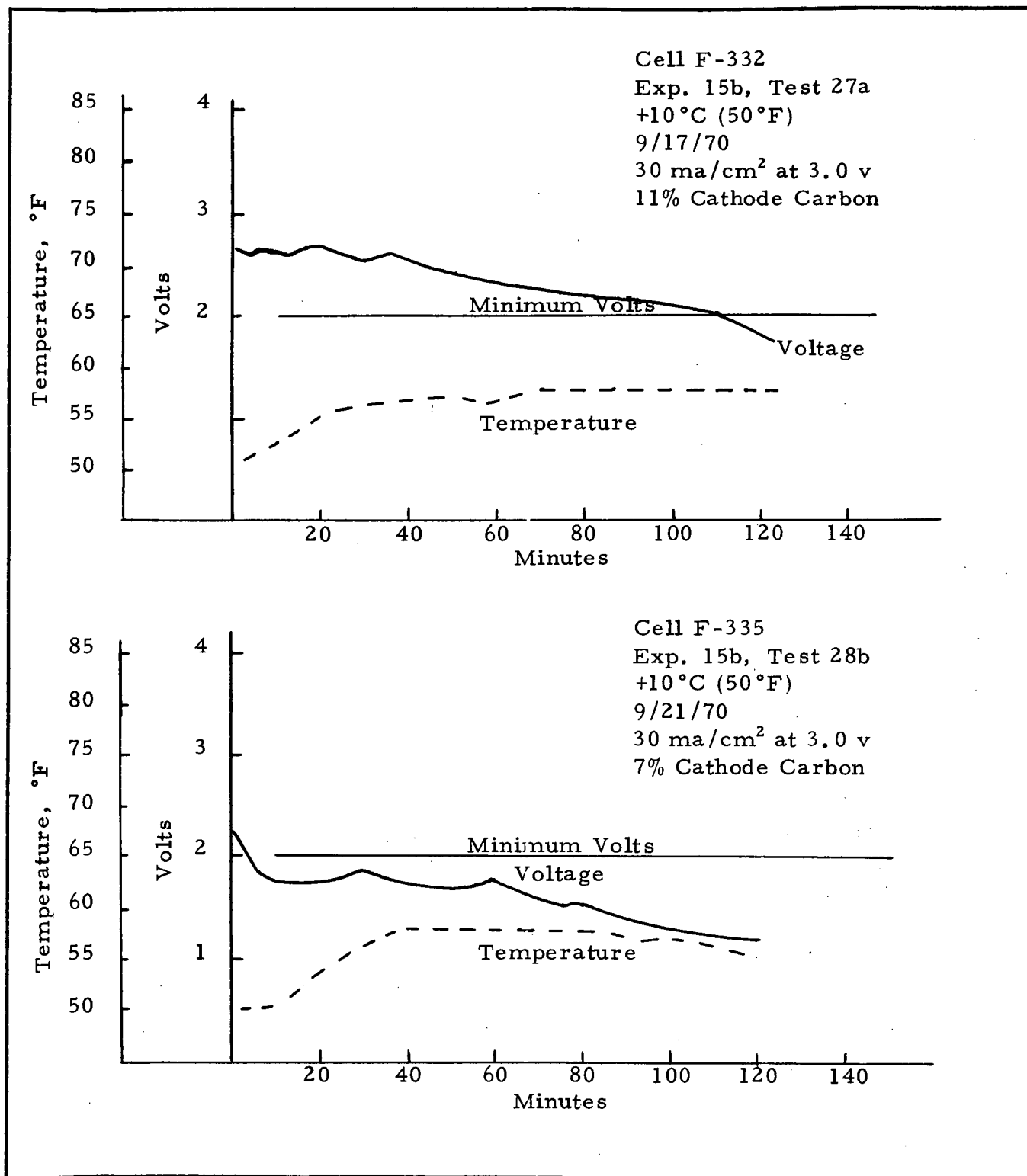
Discharge Performance of Cells in Experiment #15b



FORM FM-100

FIGURE 36

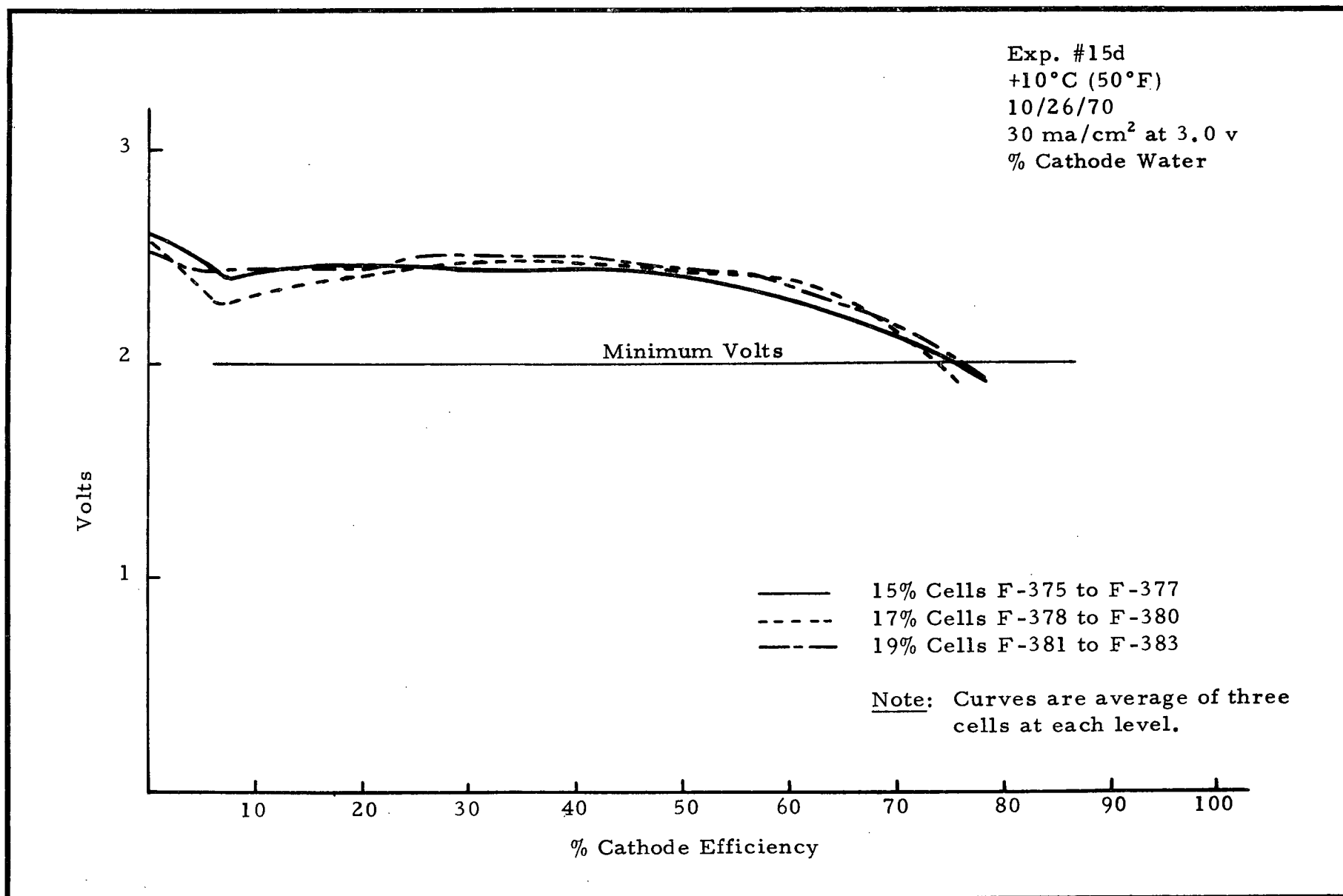
Discharge Performance of Cells in Experiment #15b



FORM FM-100

FIGURE 37

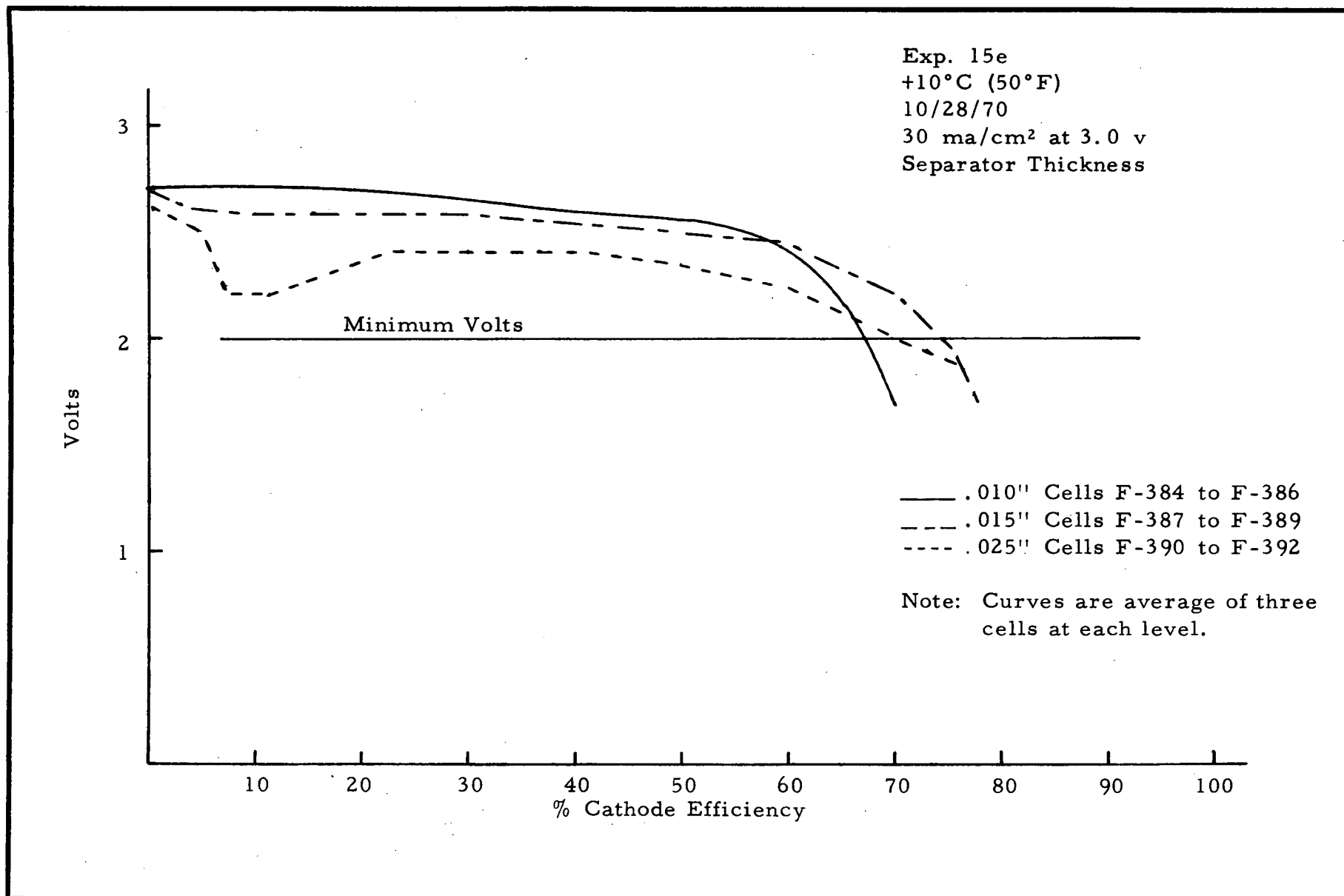
Discharge Performance of Cells in Experiment #15b



FORM FM-101

FIGURE 38

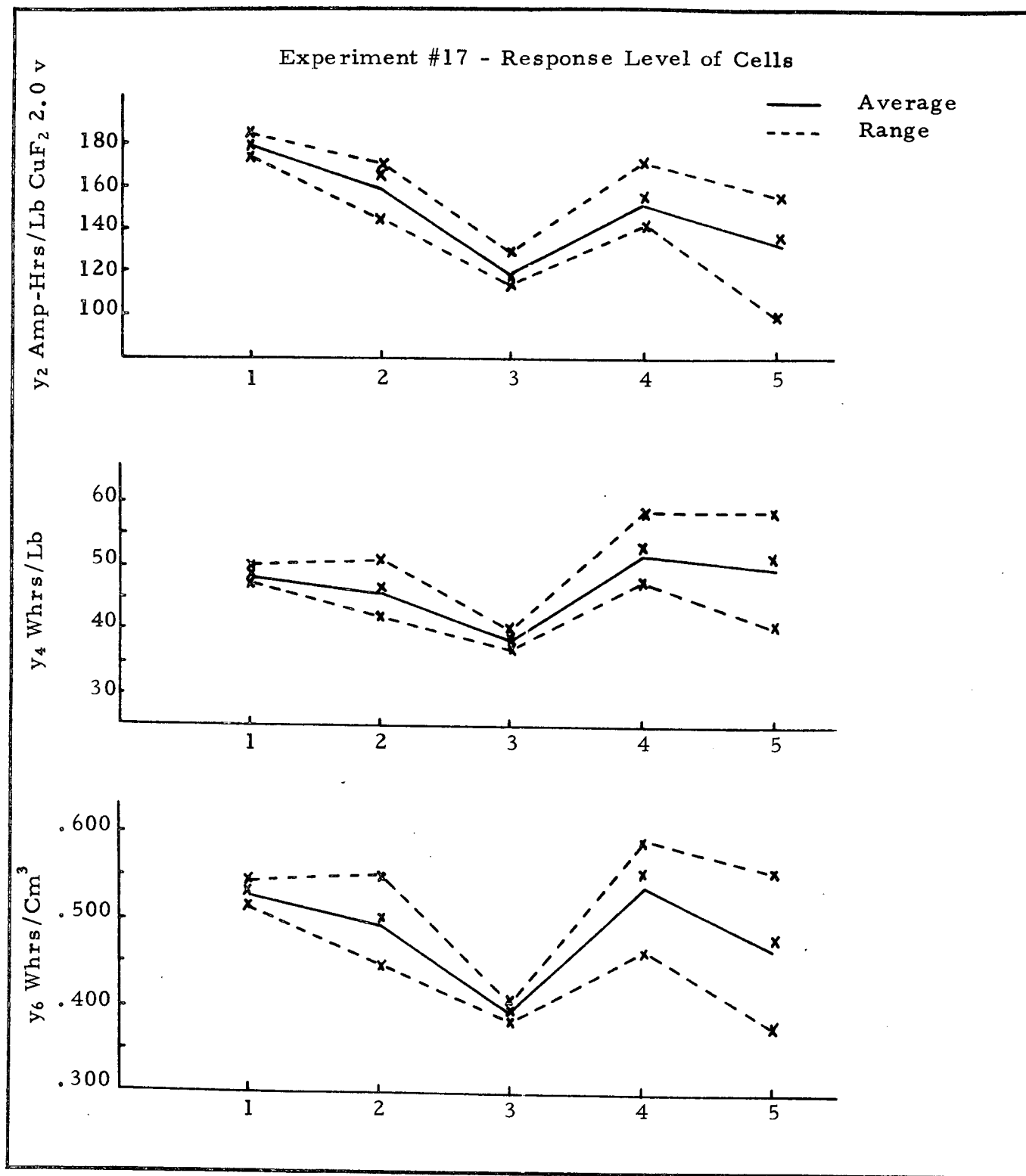
Discharge Performance of Cells in Experiment #15d



FORM FM-101

FIGURE 39

Discharge Performance of Cells in Experiment #15e

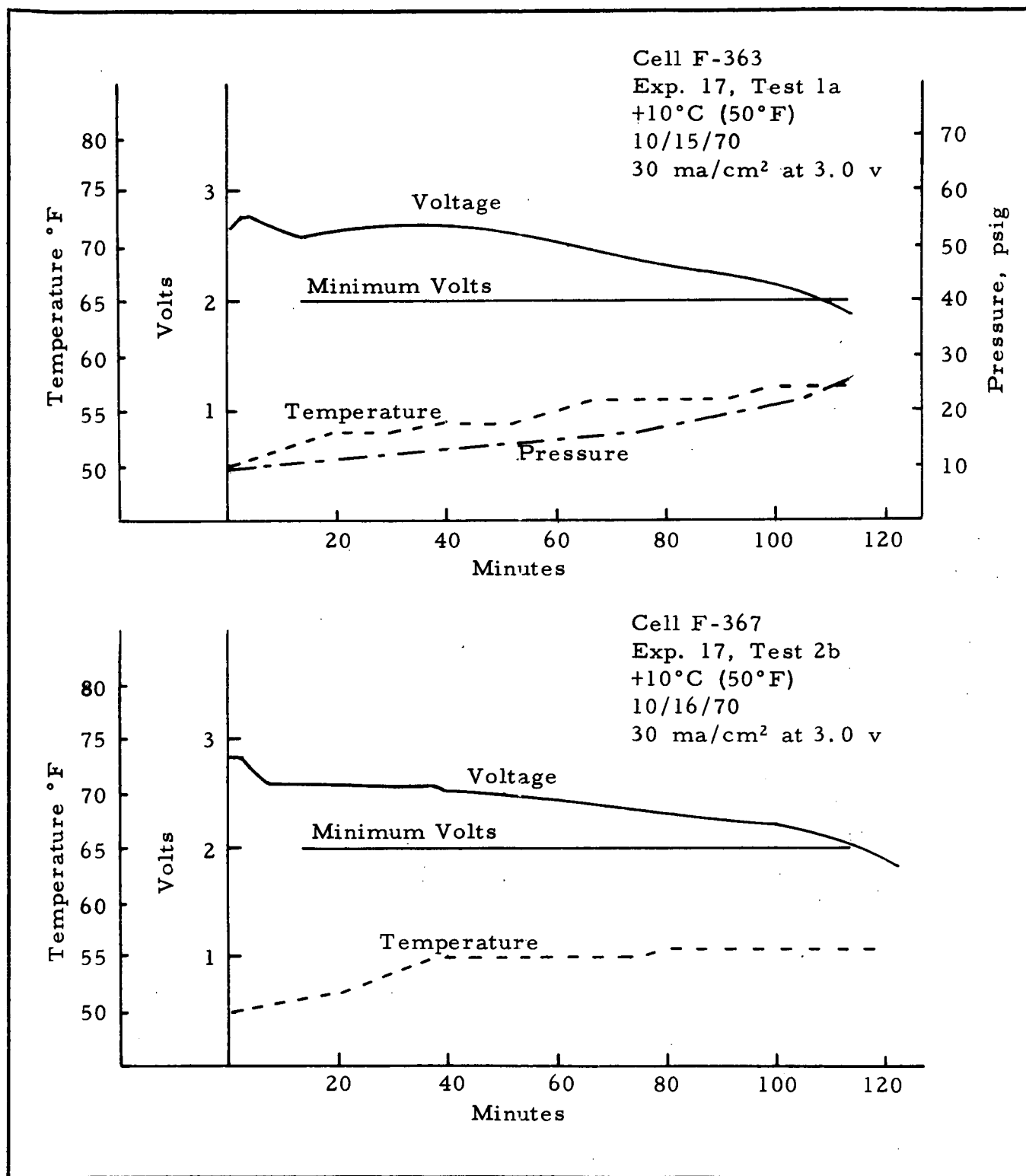


FORM FM-100

FIGURE 40

Response Level of Cells - Experiment #17





FORM FM-100

FIGURE 41

Discharge Performance of Cells in Experiment #17

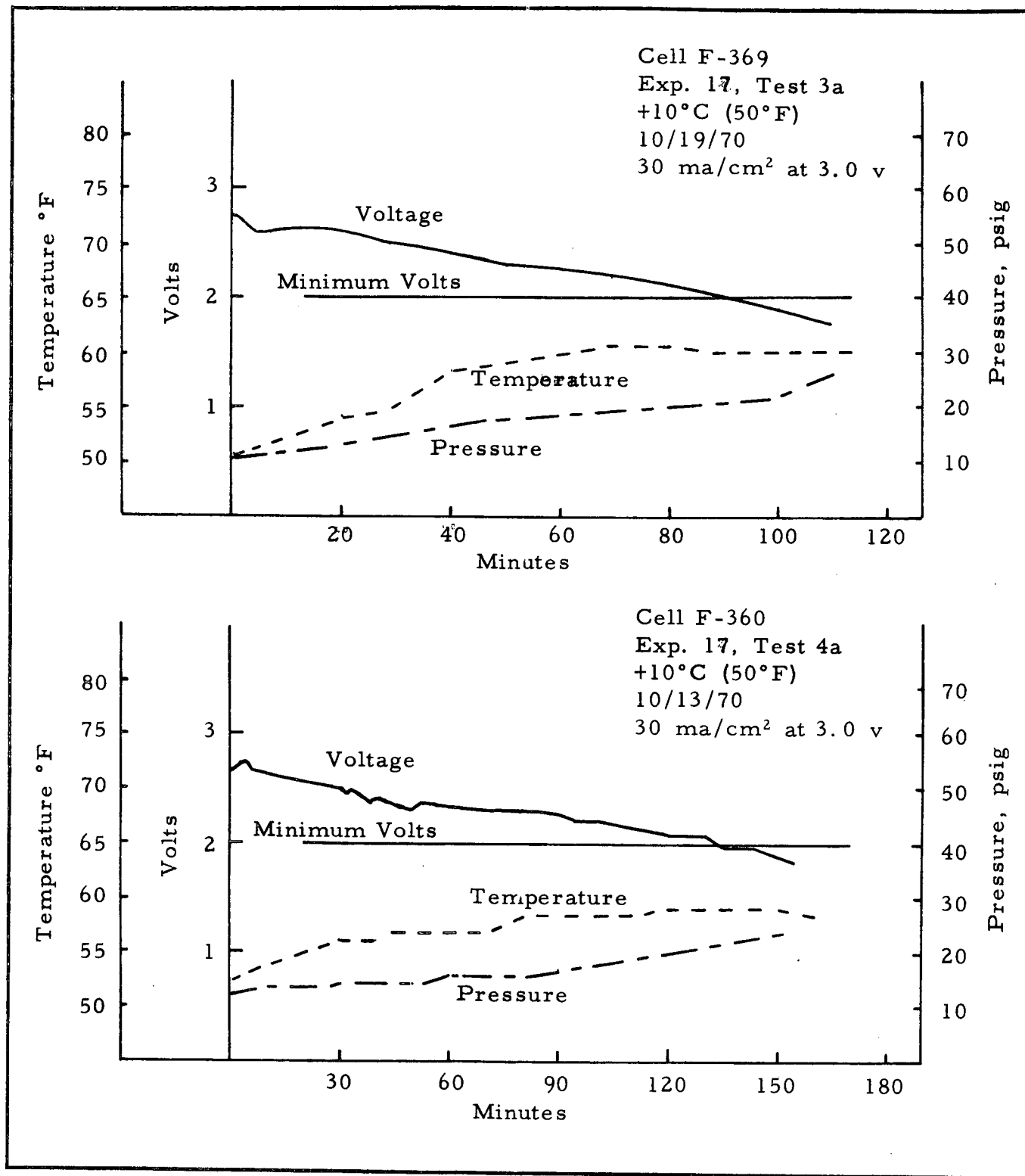


FIGURE 42

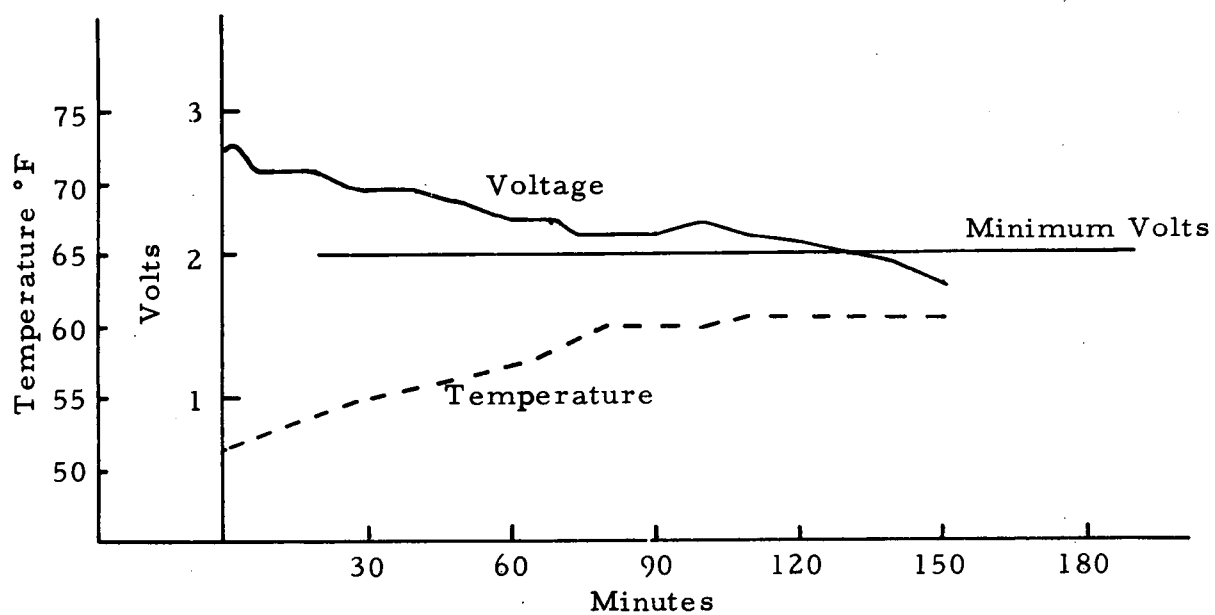
Discharge Performance of Cells in Experiment #17

Cell F-374

Exp. 17, Test 5c

+10°C (50°F)

10/20/70

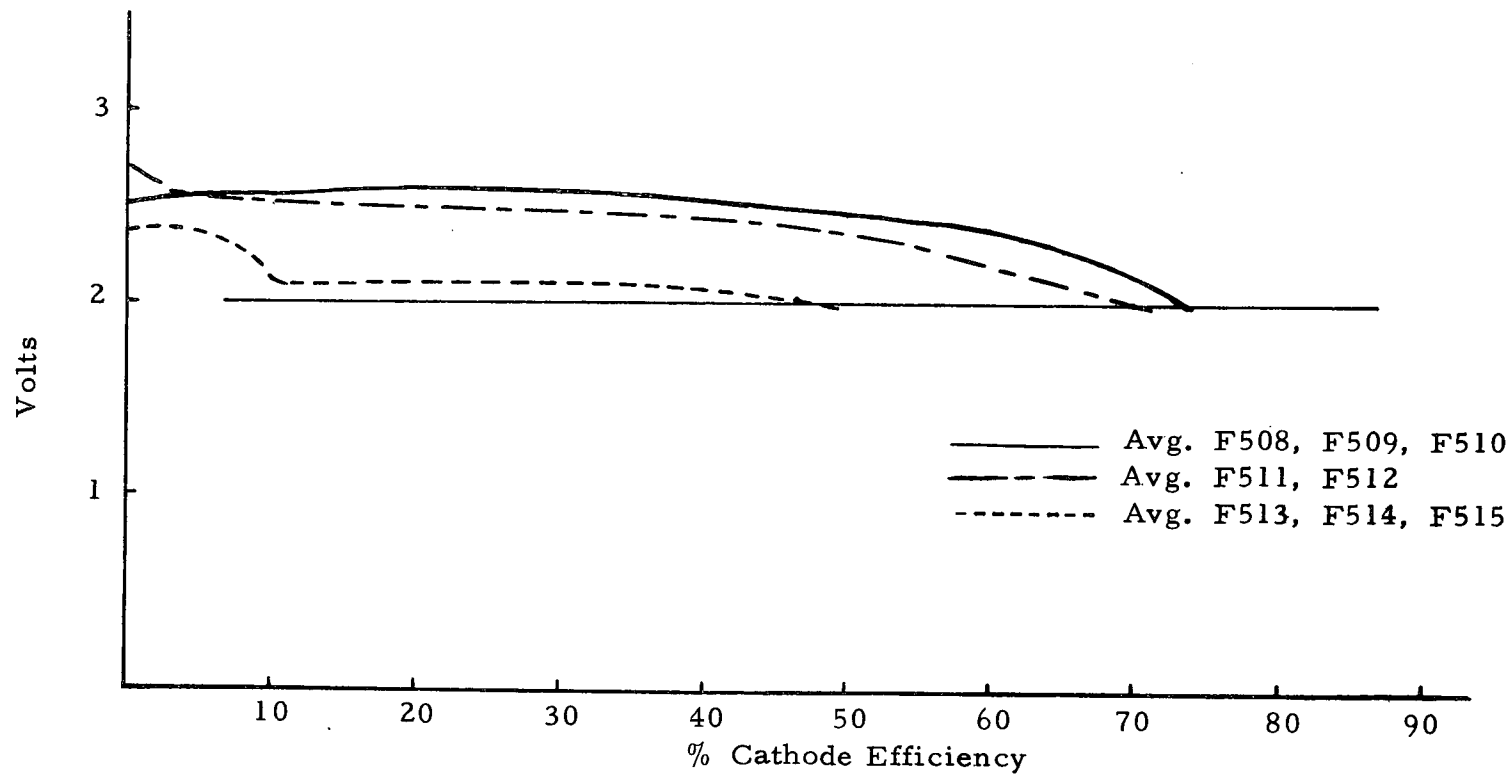
30 ma/cm<sup>2</sup> at 3.0 v

FORM FM-100

FIGURE 43

Discharge Performance of Cells in Experiment #17

Experiment #24  
Vector Performance  
+10°C  
30 ma/cm<sup>2</sup>

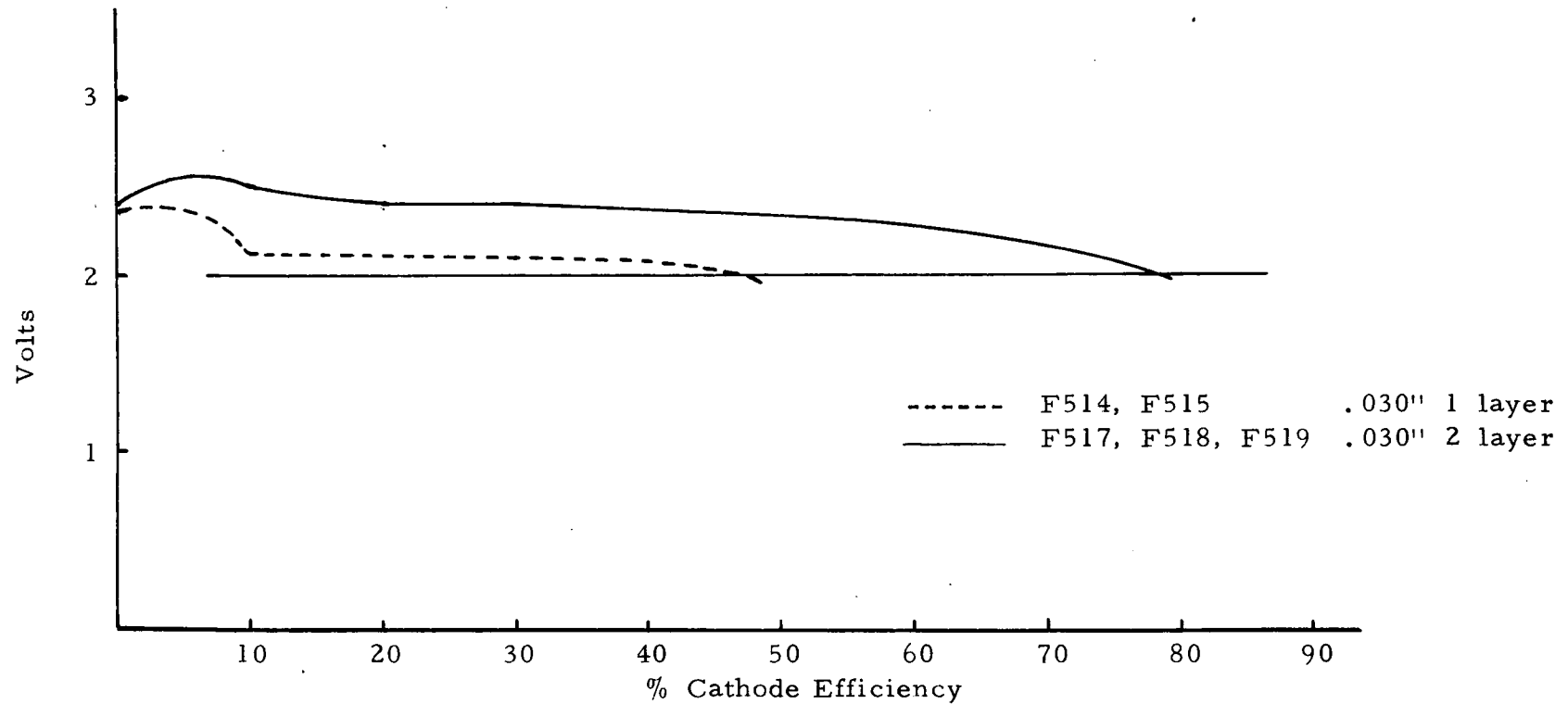


FORM FM-101

FIGURE 44

Vector Performance - Experiment #24

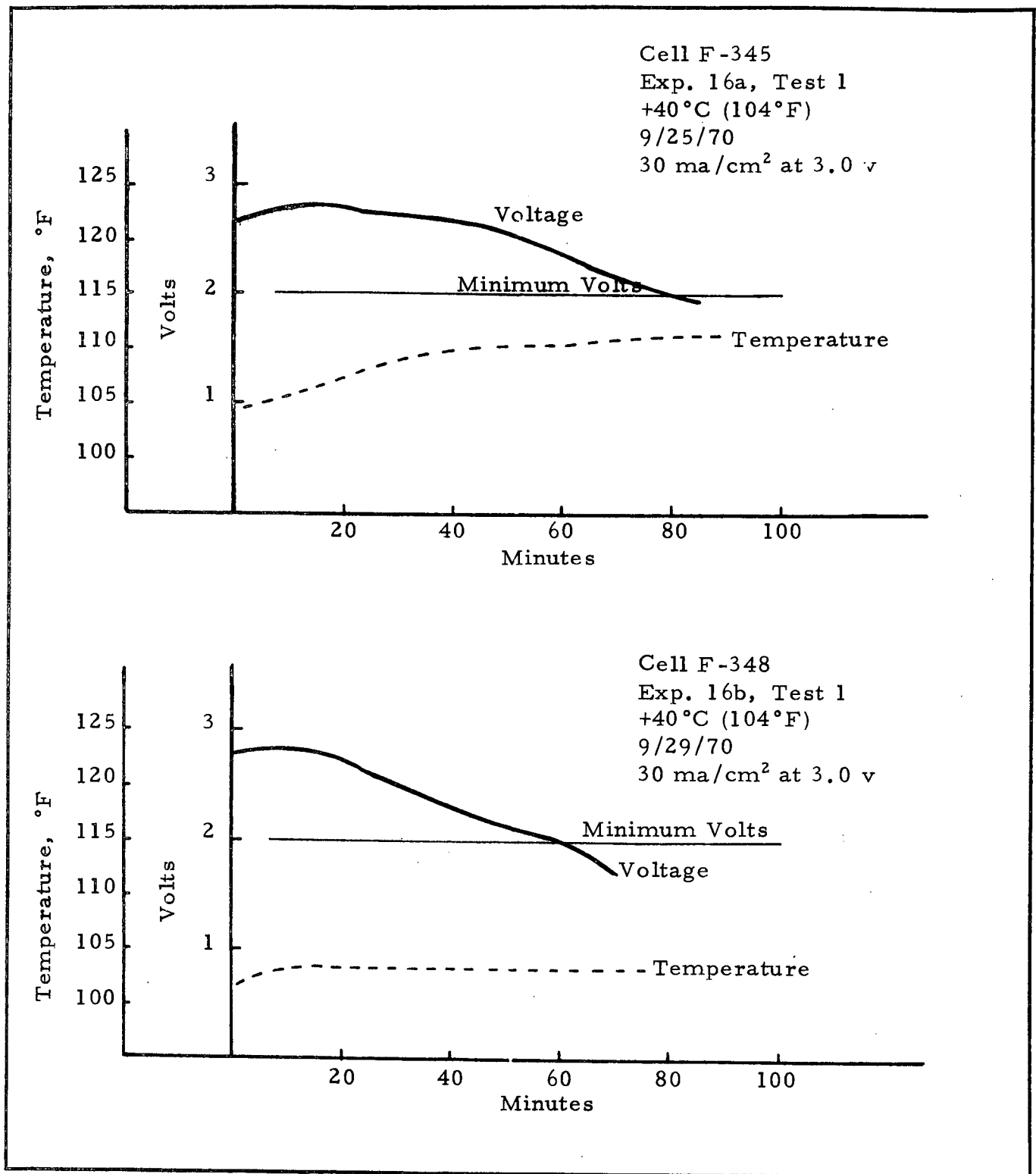
Experiment #24  
Effect of Separator Layers  
+10°C  
30 ma/cm<sup>2</sup>



FORM FM-101

FIGURE 45

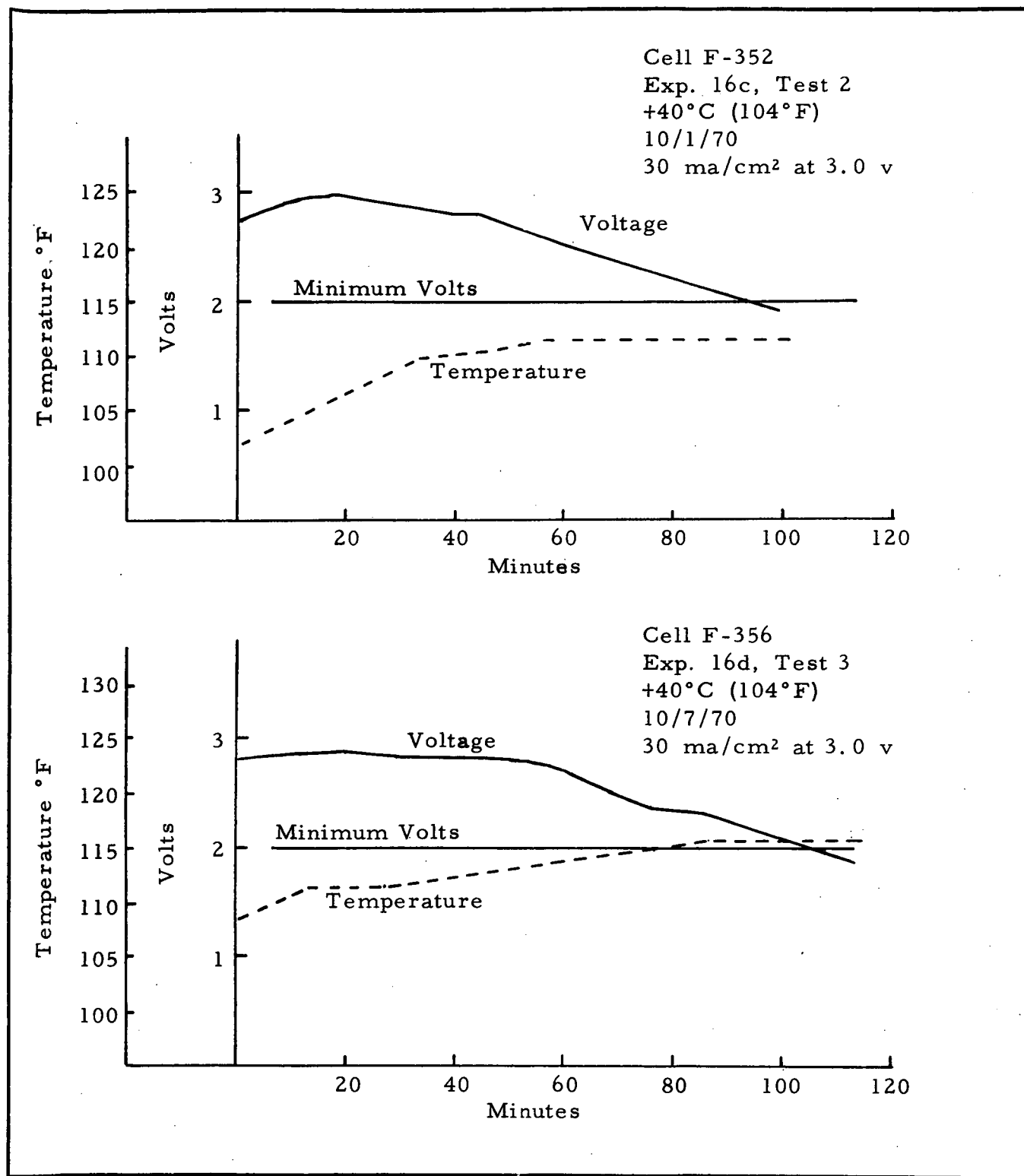
Effect of Separator Layers - Experiment #24



FORM FM-100

FIGURE 46

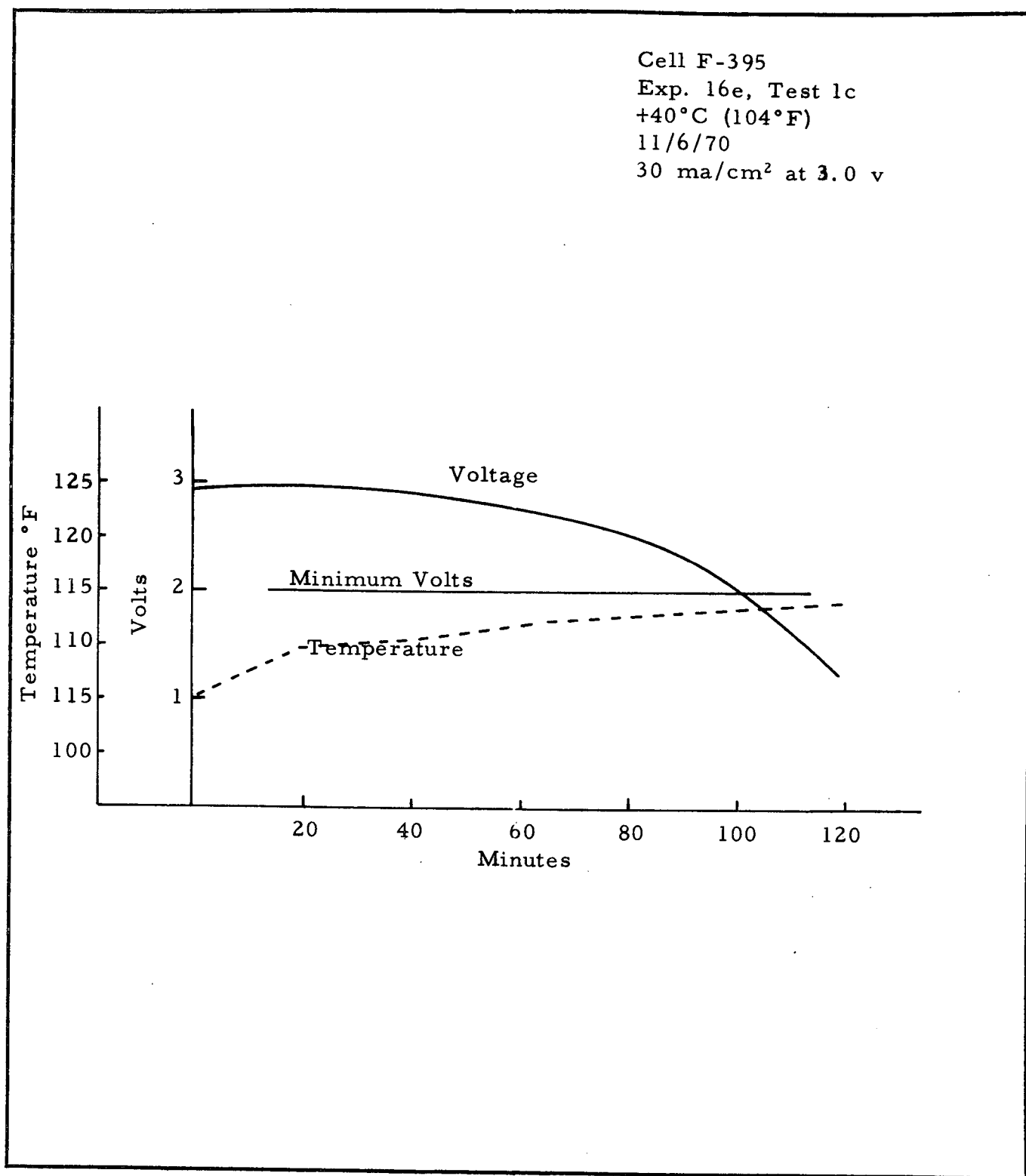
Discharge Performance of Cells in Experiments #16a and #16b



FORM FM-100

FIGURE 47

Discharge Performance of Cells in Experiments #16c and #16d



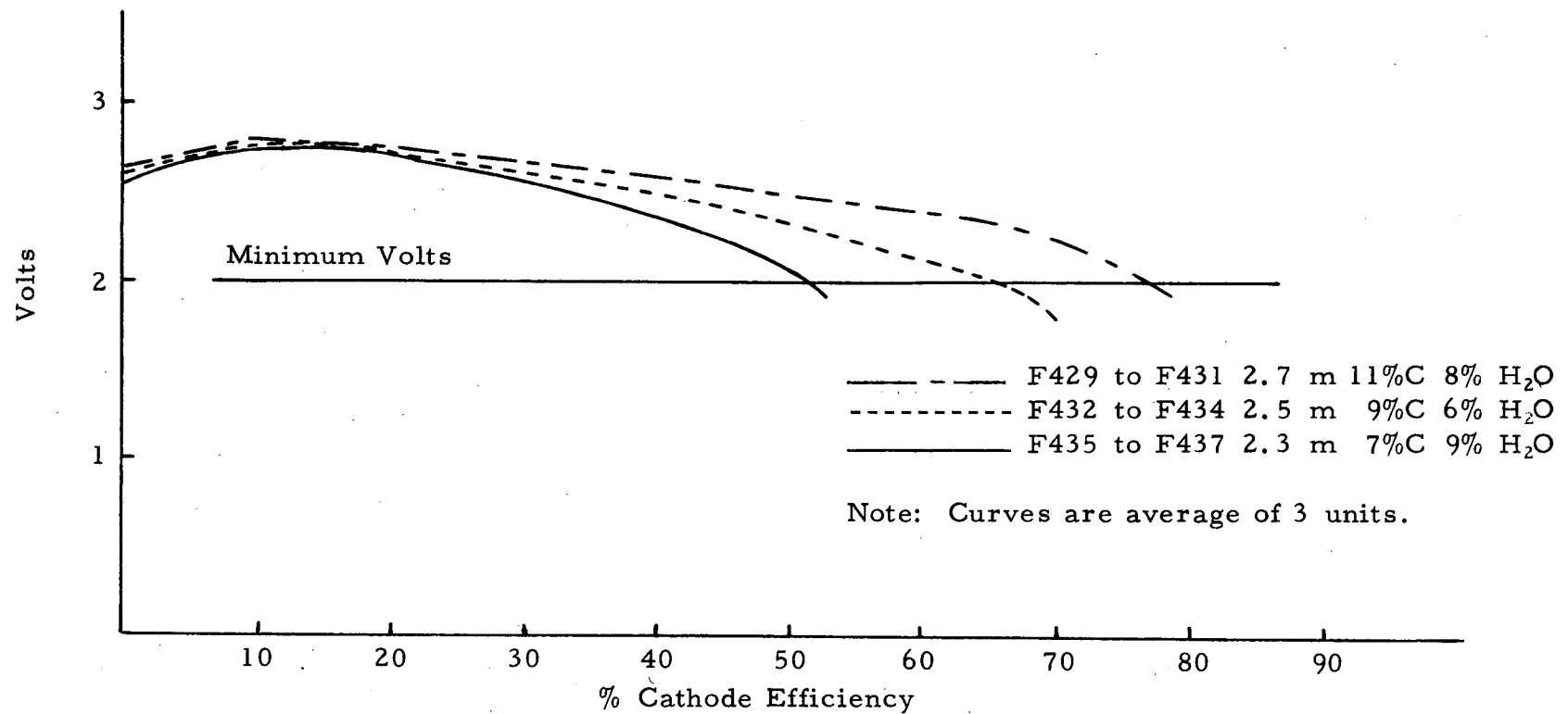
FORM FM-100

FIGURE 48

Discharge Performance of Cells in Experiment #16e



Exp. 19a  
 +10°C (50°F)  
 12/4/70  
 20 ma/cm<sup>2</sup> at 2.5 v

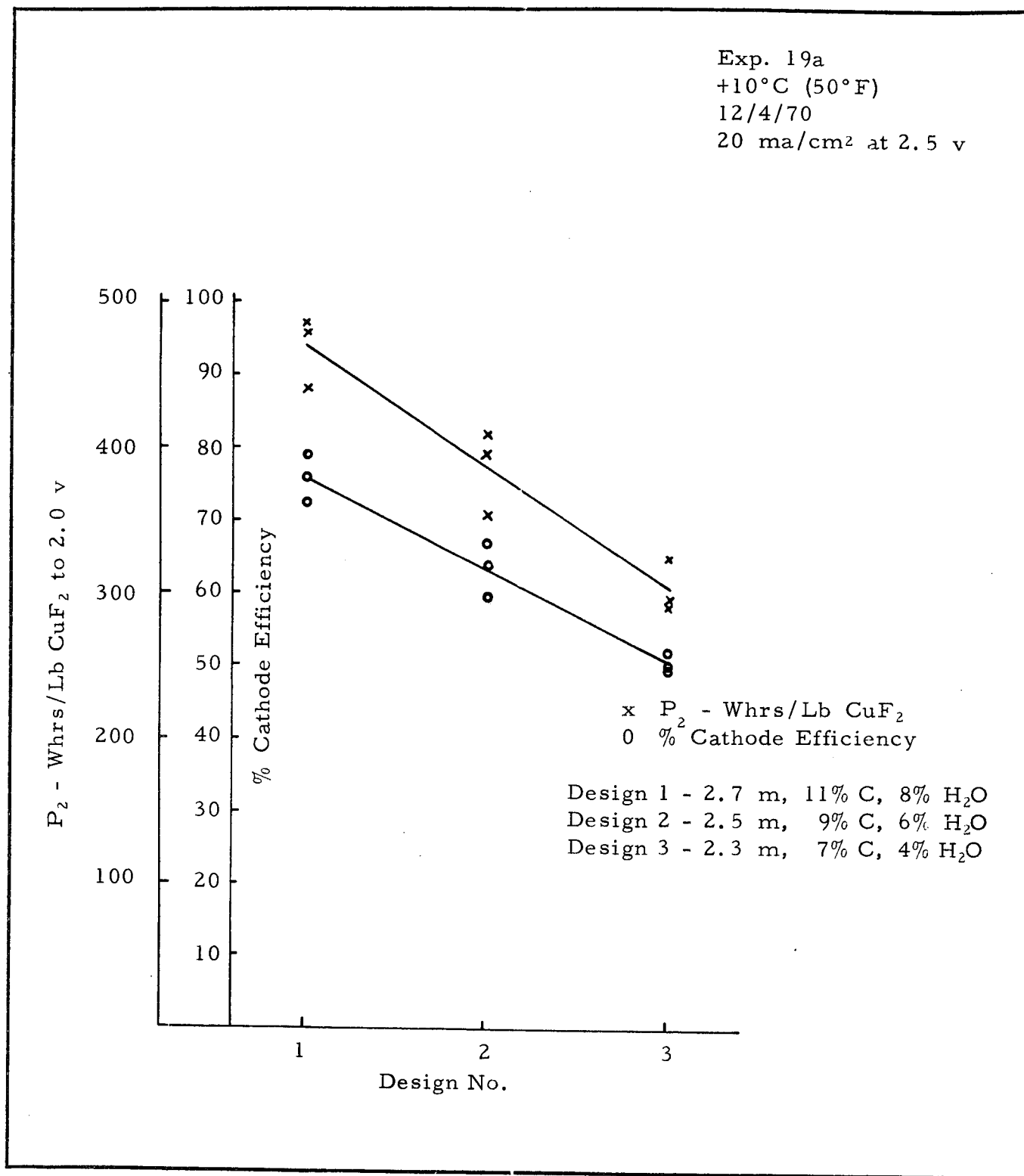


FORM FM-101

FIGURE 49

Discharge Performance of Cells in Experiment #19a

## Experiment #19a - Response Levels



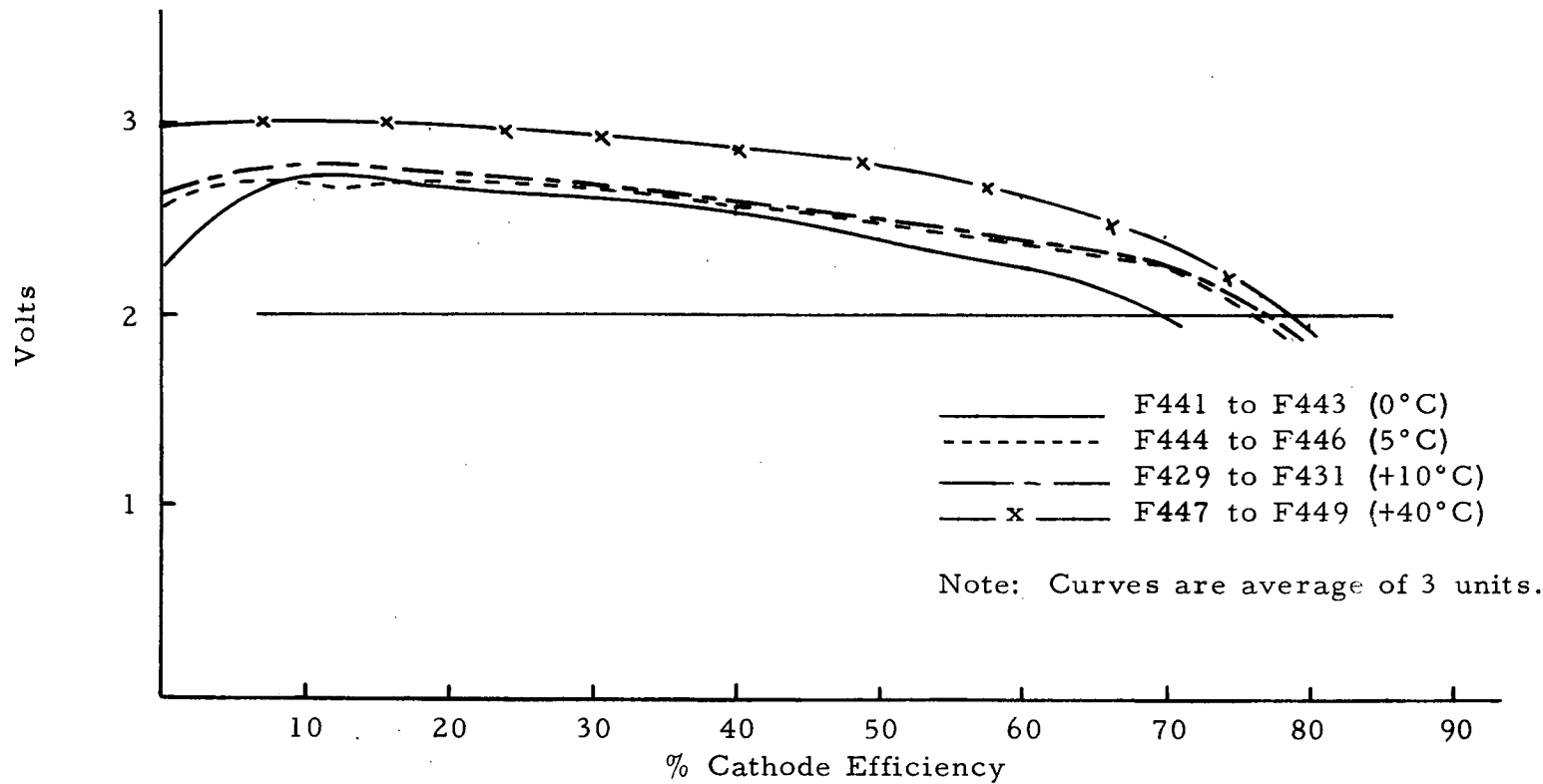
FORM FM-100

FIGURE 50

Response Levels - Experiment #19a

Exp. 19b  
12/21/70  
20 ma/cm<sup>2</sup> at 2.5 v

### Temperature Scan



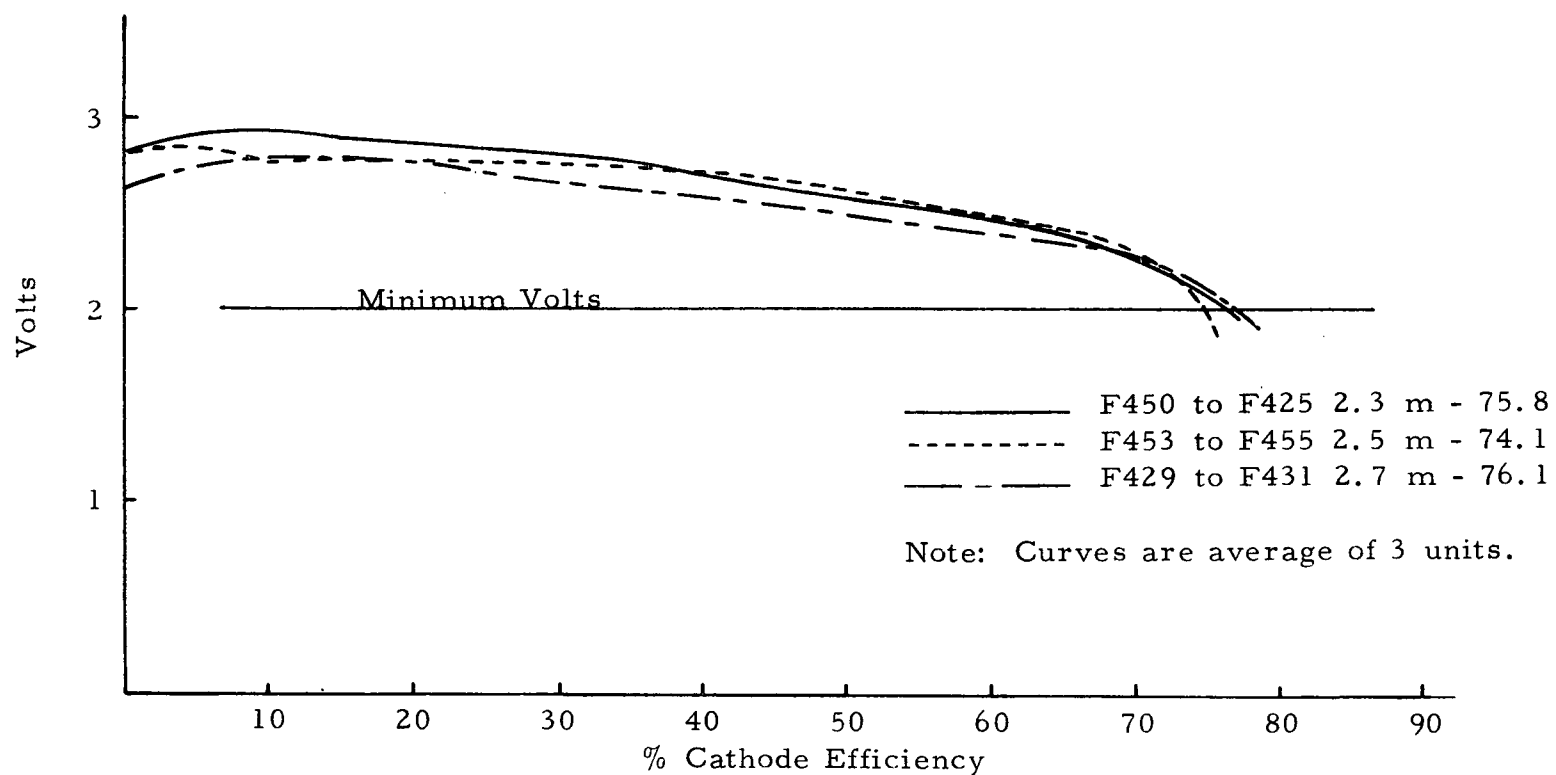
FORM FM-101

FIGURE 51

Discharge Performance of Cells in Experiment #19b

Exp. 19c  
+10°C (50°F)  
12/28/70  
20 ma/cm<sup>2</sup> at 2.5 v

Electrolyte Molarity

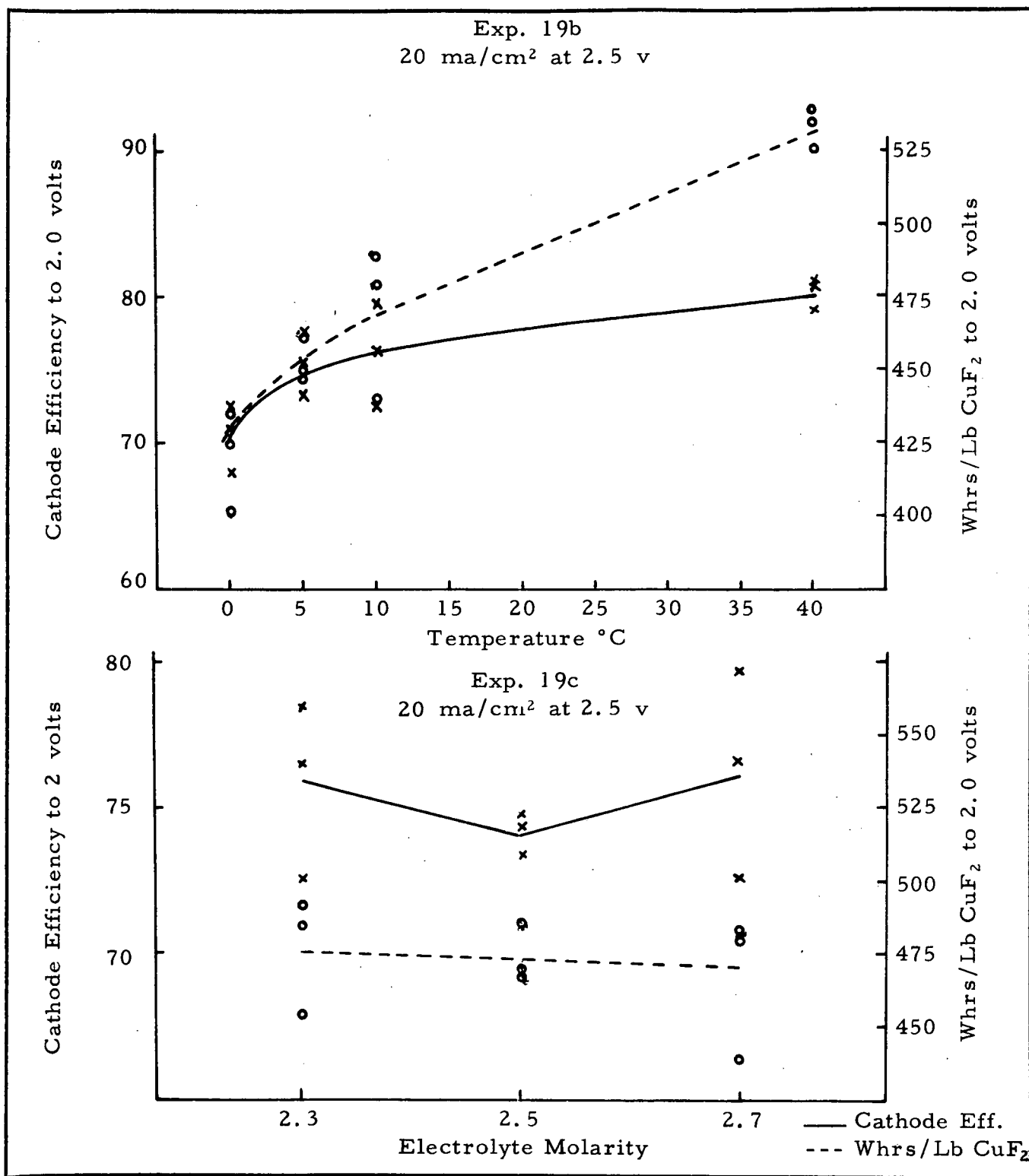


FORM FM-101

FIGURE 52

Discharge Performance of Cells in Experiment #19c

## Experiments #19b and #19c - Response Levels

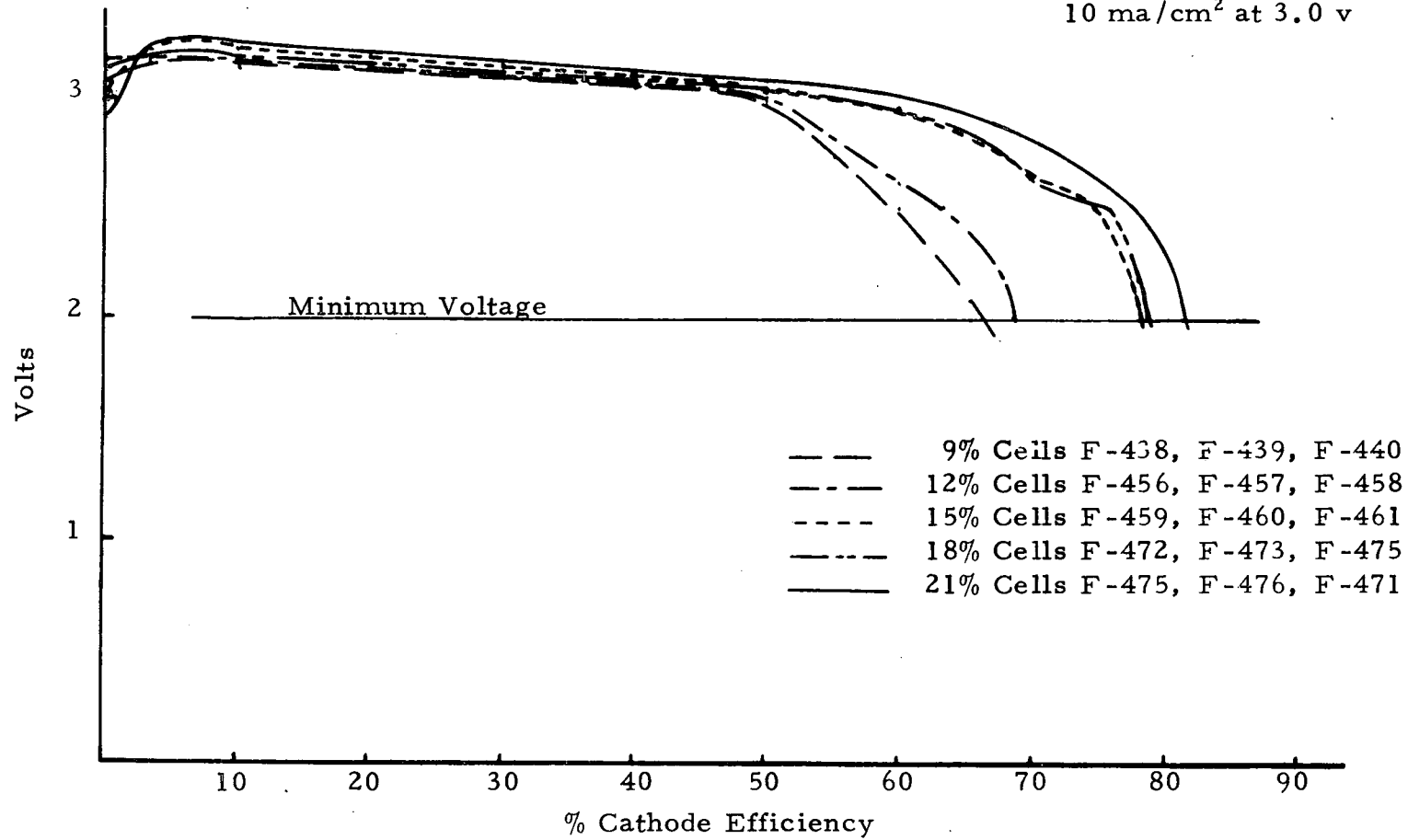


FORM FM-100

FIGURE 53

Response Levels - Experiments #19b and #19c

Experiment 20B  
 Effect of Carbon  
 Anhydrous Cells  
 +40°C  
 10 ma/cm<sup>2</sup> at 3.0 v

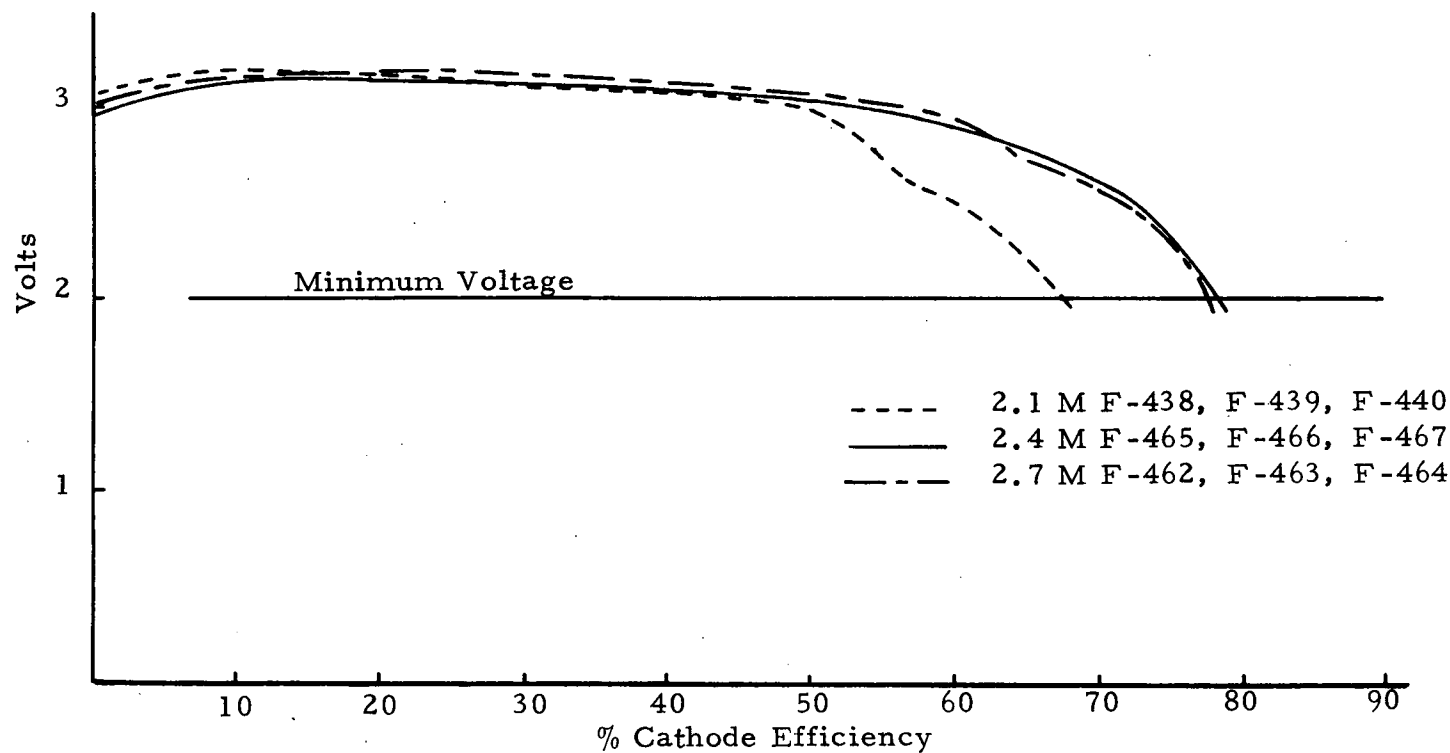


FORM FM-101

FIGURE 54

Discharge Performance of Cells in Experiment #20b

Experiment 20C  
 Effect of Electrolyte Concentration  
 Anhydrous Cells  
 +40 °C  
 10 ma/cm<sup>2</sup> at 3.0 v

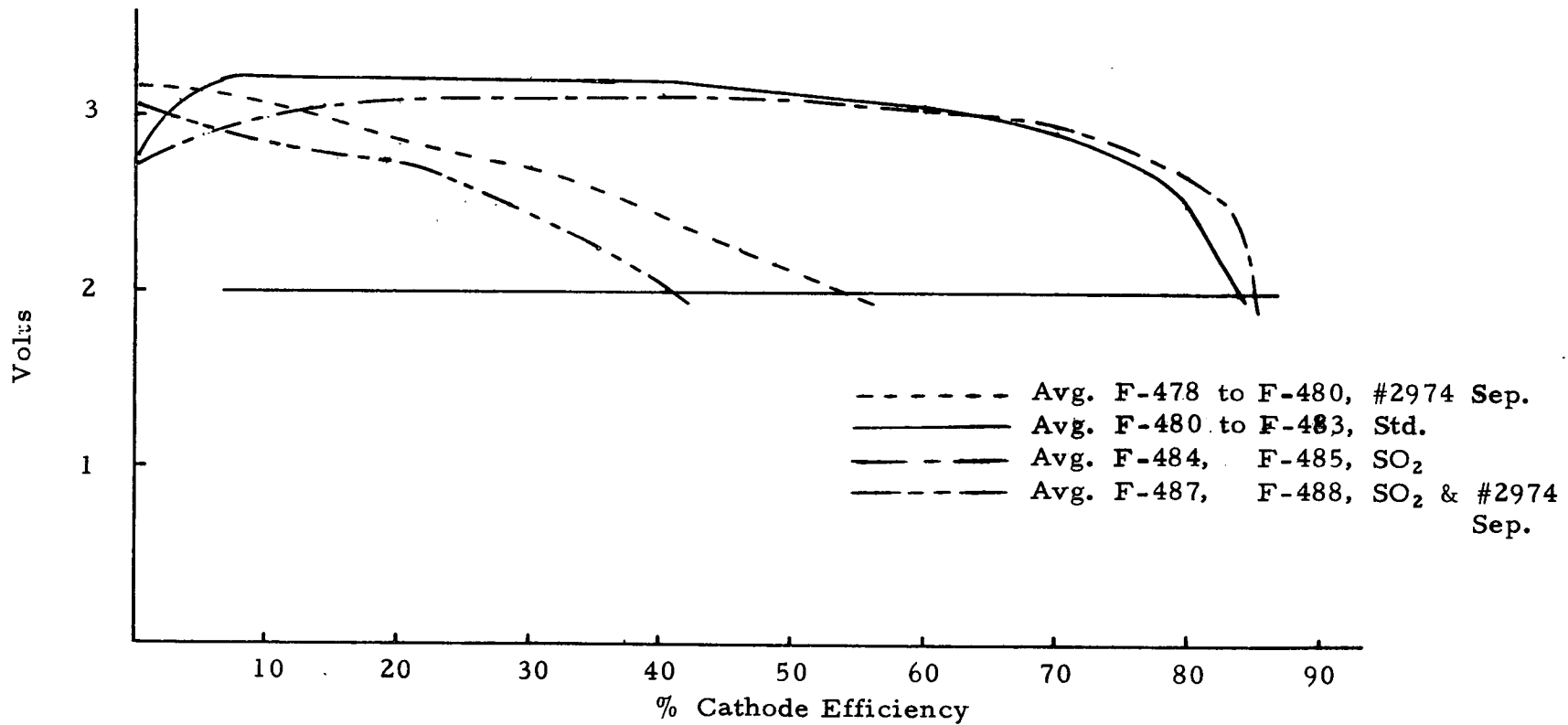


FORM FM-101

FIGURE 55

Discharge Performance of Cells in Experiment #20c

Exp. 21  
+40° C  
10 ma/cm<sup>2</sup> at 3.0 v



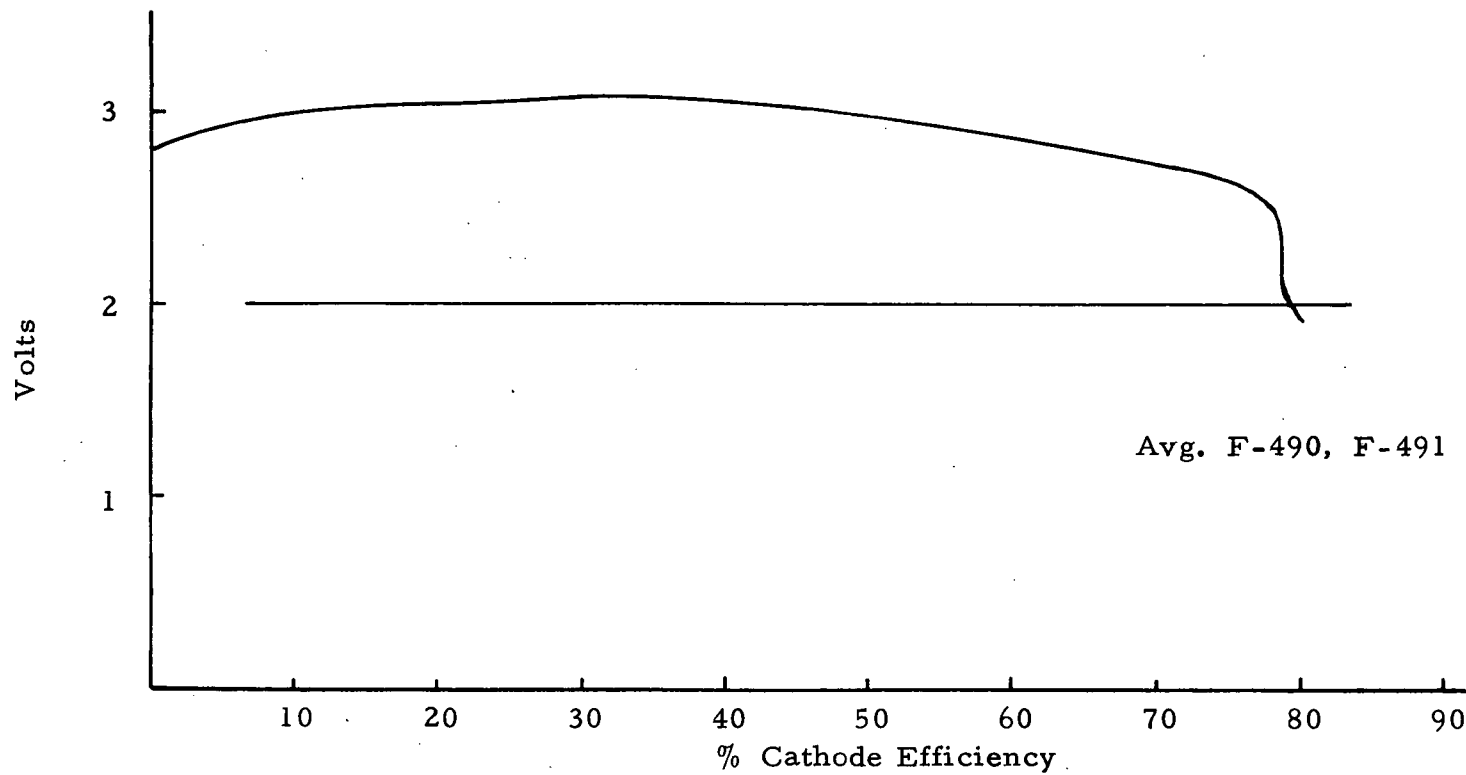
FORM FM-101

FIGURE 56

Discharge Performance of Cells in Experiment #21



Exp. 22a  
2.5 M LiBF<sub>4</sub>/MF  
5 psig SO<sub>2</sub>  
+40°C  
10 ma/cm<sup>2</sup> at 3.0 v

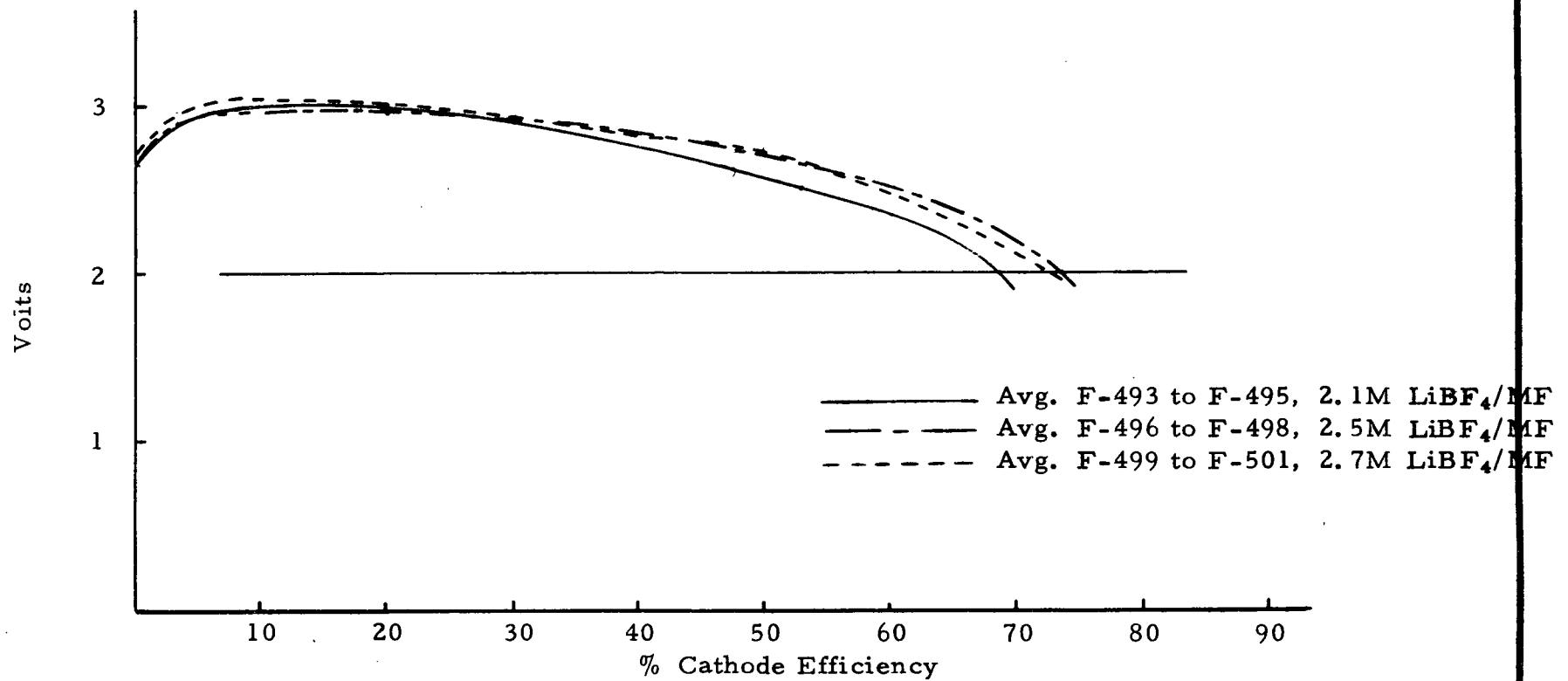


FORM FM-101

FIGURE 57

Discharge Performance of Cells in Experiment #22a

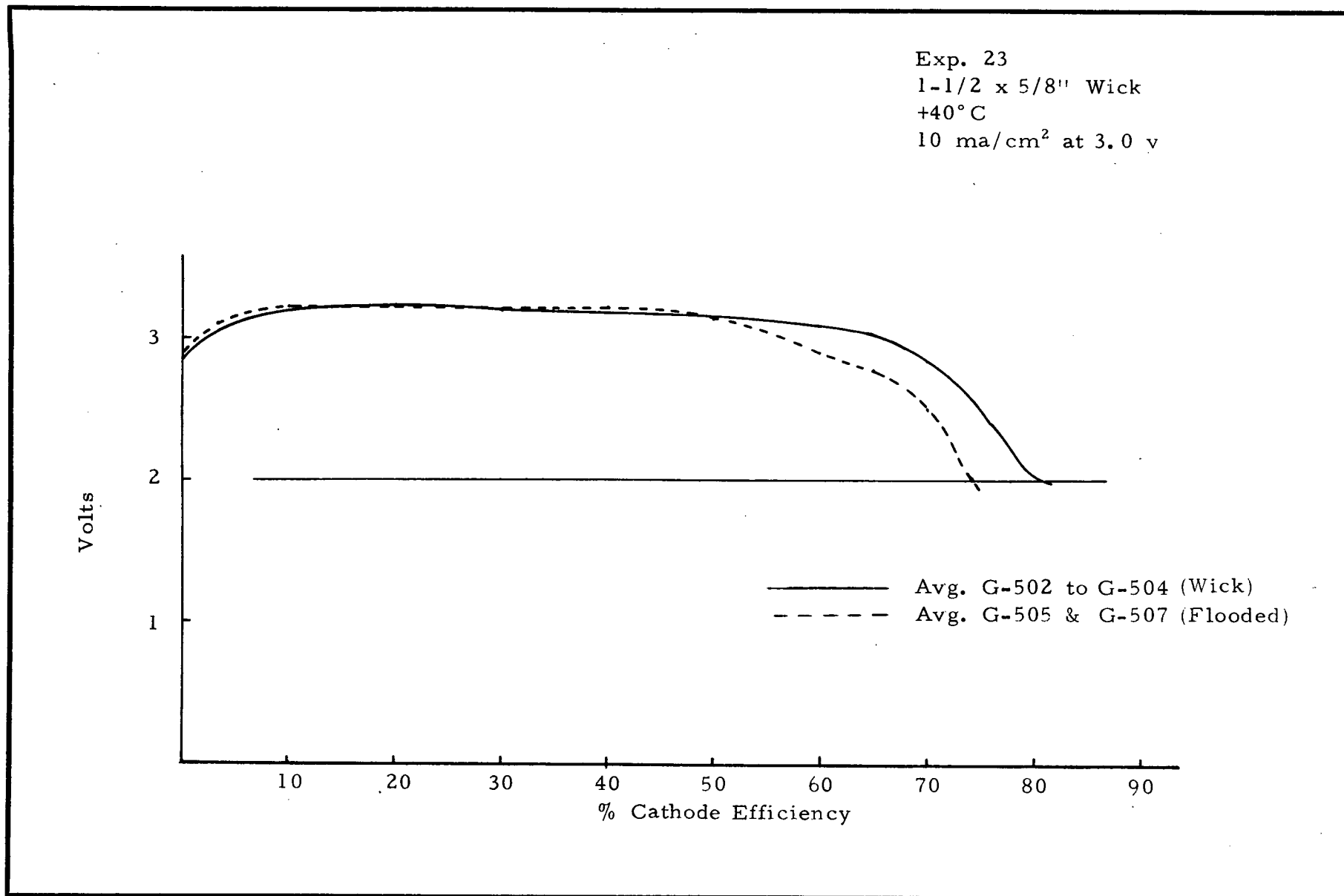
Exp. 22b  
Effects of  $\text{LiBF}_4/\text{MF}$   
 $+40^\circ\text{C}$   
 $10\text{ ma/cm}^2$  at  $3.0\text{ v}$



FORM FM-101

FIGURE 58

Discharge Performance of Cells in Experiment #22b

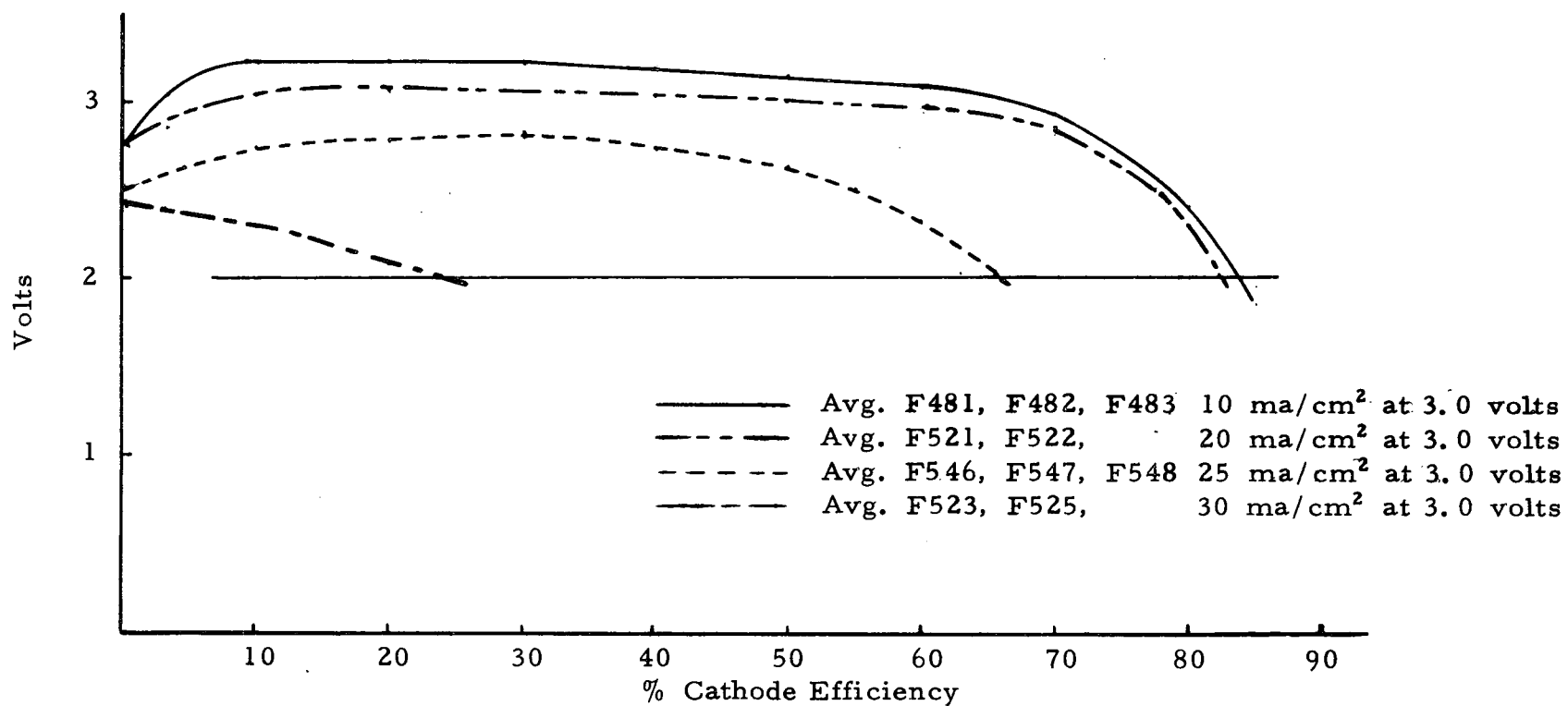


FORM FM-101

FIGURE 59

Discharge Performance of Cells in Experiment #23

Experiment #26a  
Effect of Discharge Rate  
+40°C

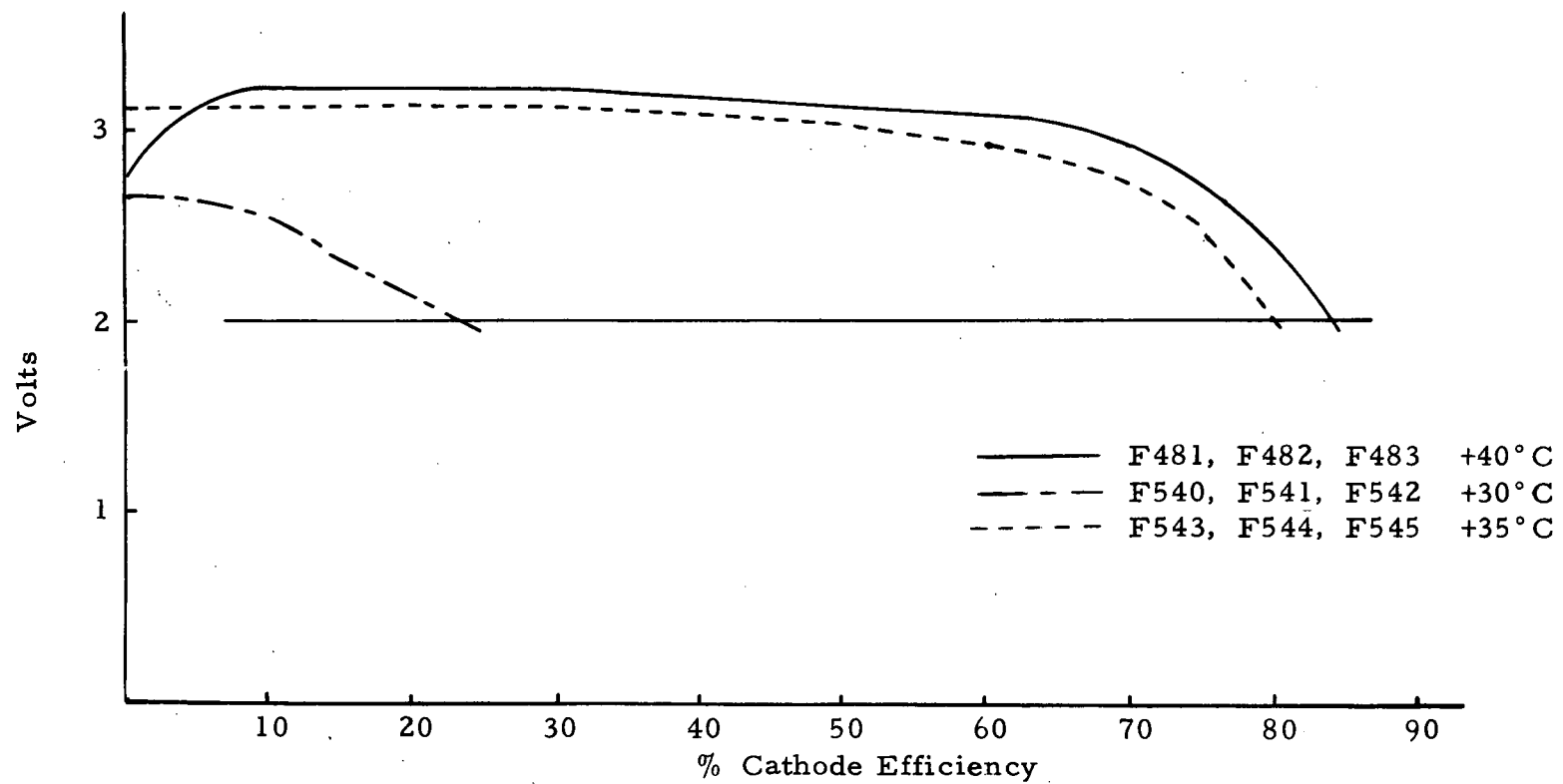


FORM FM-101

FIGURE 60

Discharge Performance of Cells in Experiment #26a

Experiment #26b  
Effect of Temperature  
10 ma/cm<sup>2</sup> at 3.0 volts



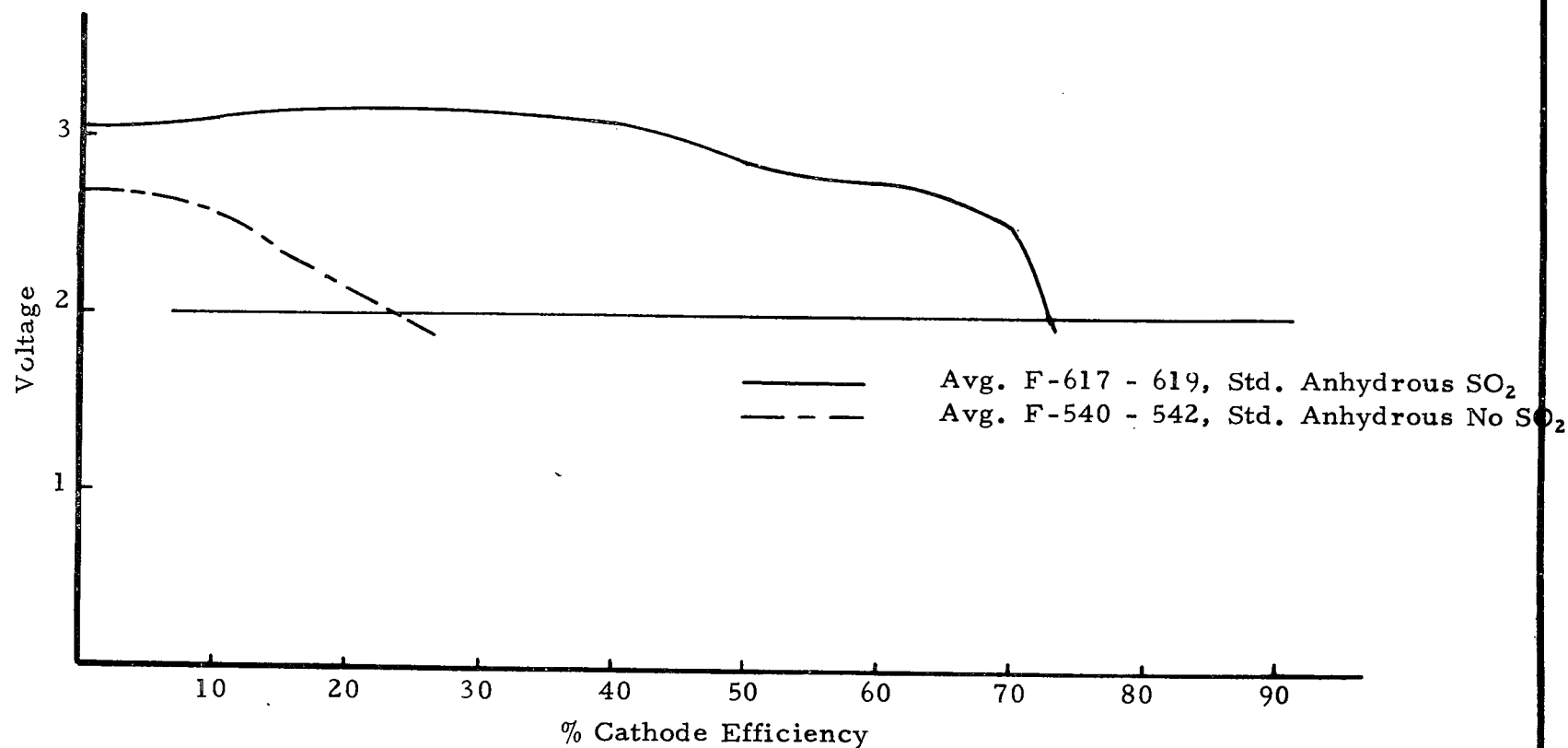
FORM FM-101

FIGURE 61

Discharge Performance of Cells in Experiment #26b

Experiment #26c  
5/18/71  
10 ma/cm<sup>2</sup> at 3.0 v  
+30°C

Effect of SO<sub>2</sub> on Lower Temperature Performance

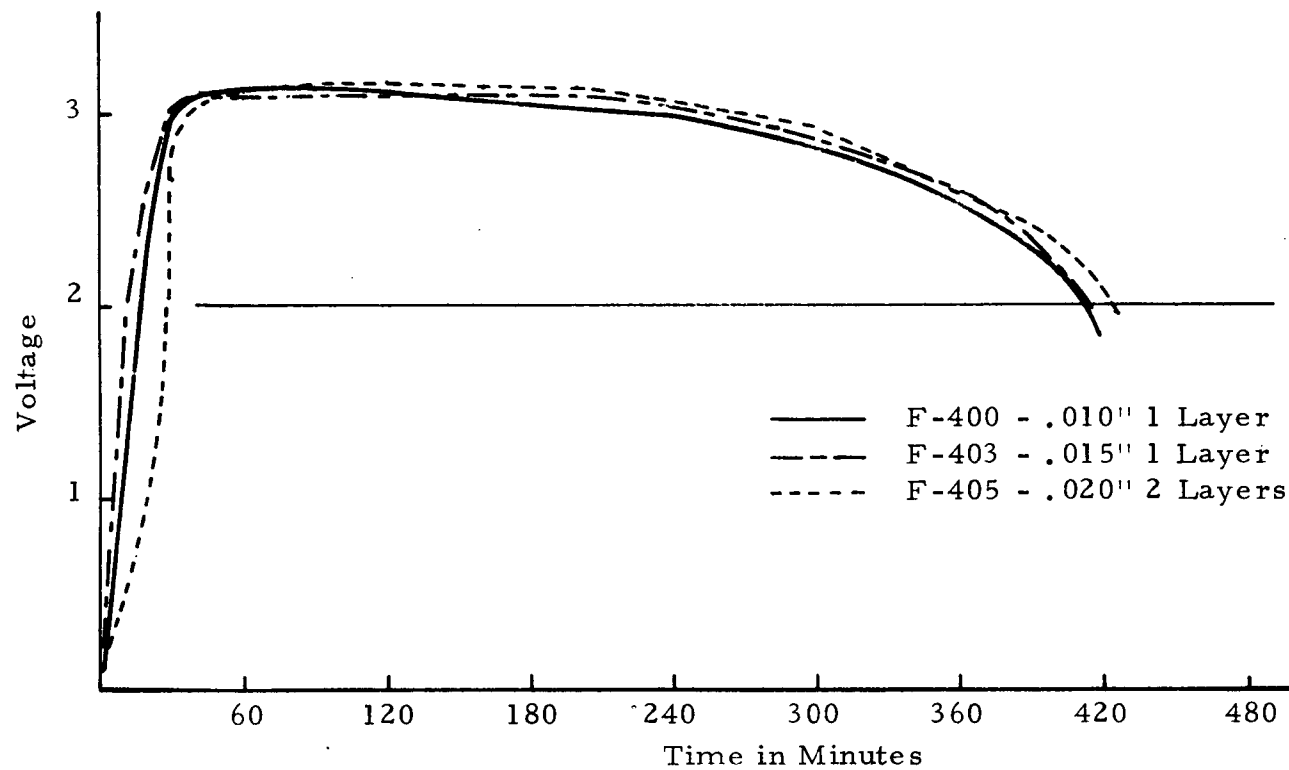


FORM FM-101

FIGURE 62

Discharge Performance of Cells in Experiment #26c

Experiment #18a  
+10°C (50°F)  
11/11/70  
10 ma/cm<sup>2</sup> at 3.0 v  
Separator Thickness

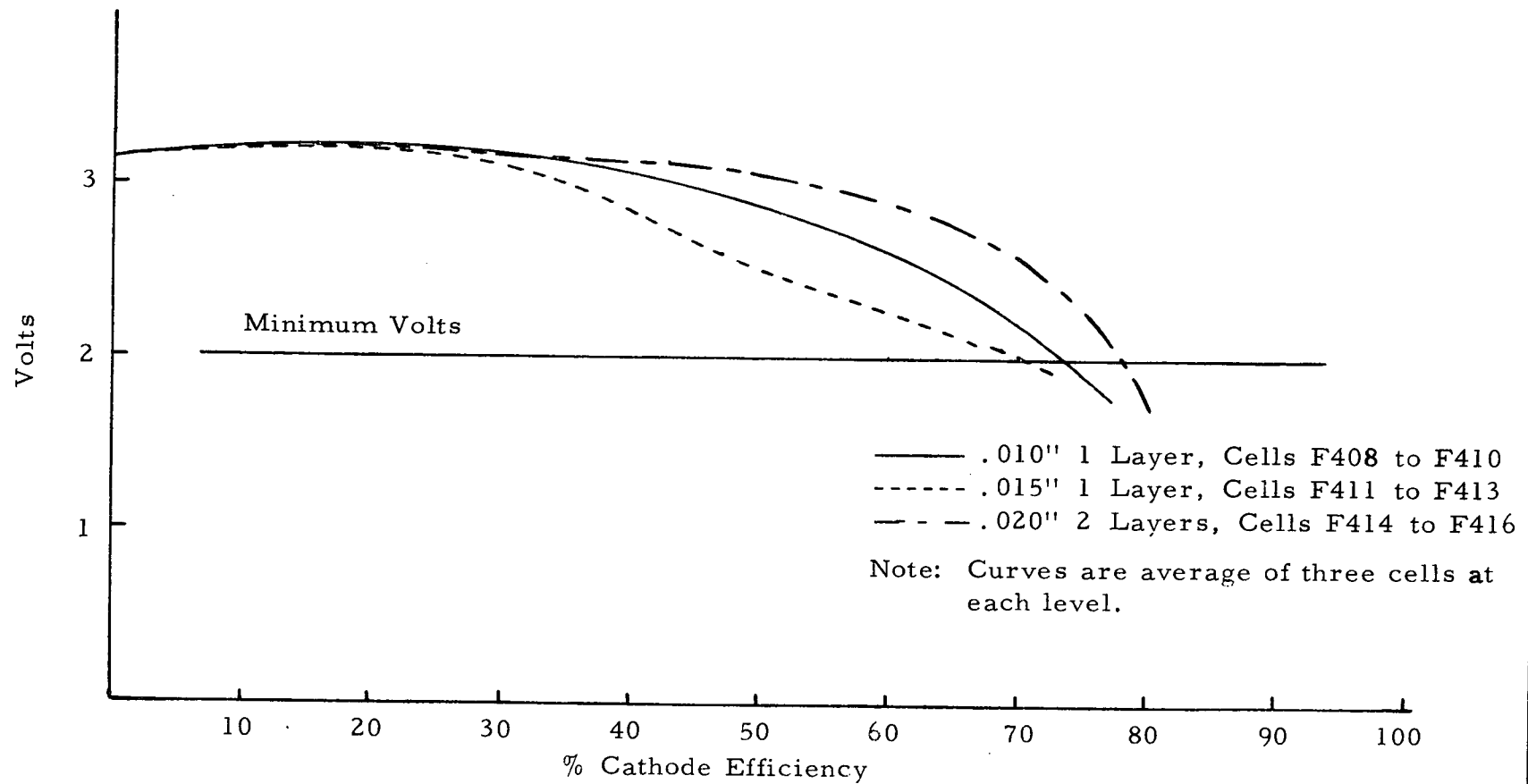


FORM FM-101

FIGURE 63

Discharge Performance of Cells in Experiment #18a

Exp. 18a  
+40°C (104°F)  
11/12/70  
10 ma/cm<sup>2</sup> at 3.0 v  
Separator Thickness



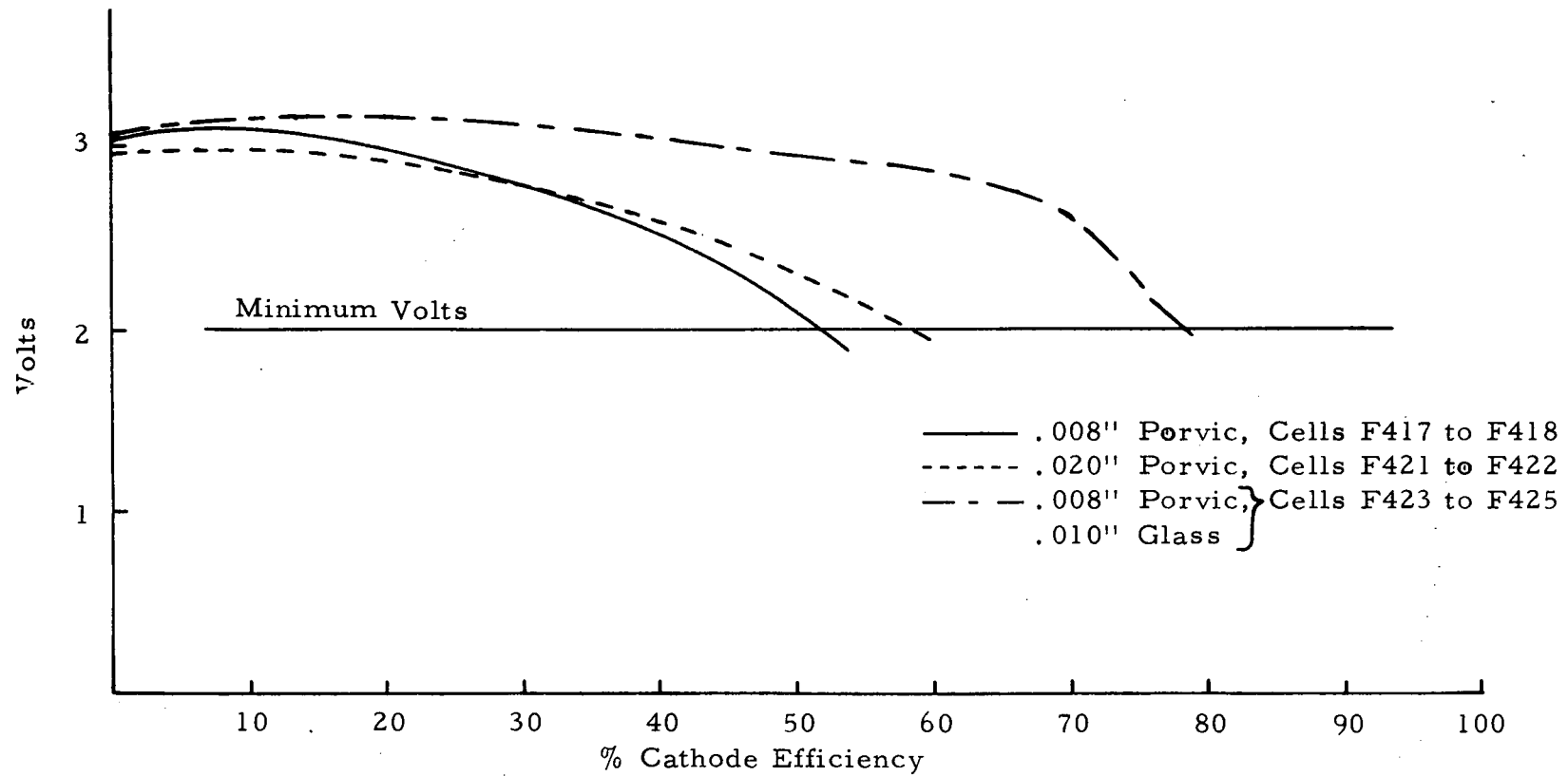
FORM FM-101

FIGURE 64

Discharge Performance of Cells in Experiment #18a



Exp. 18c  
 +40°C (104°F)  
 11/23/70  
 10 ma/cm<sup>2</sup> at 3.0 v  
 Separator Thickness

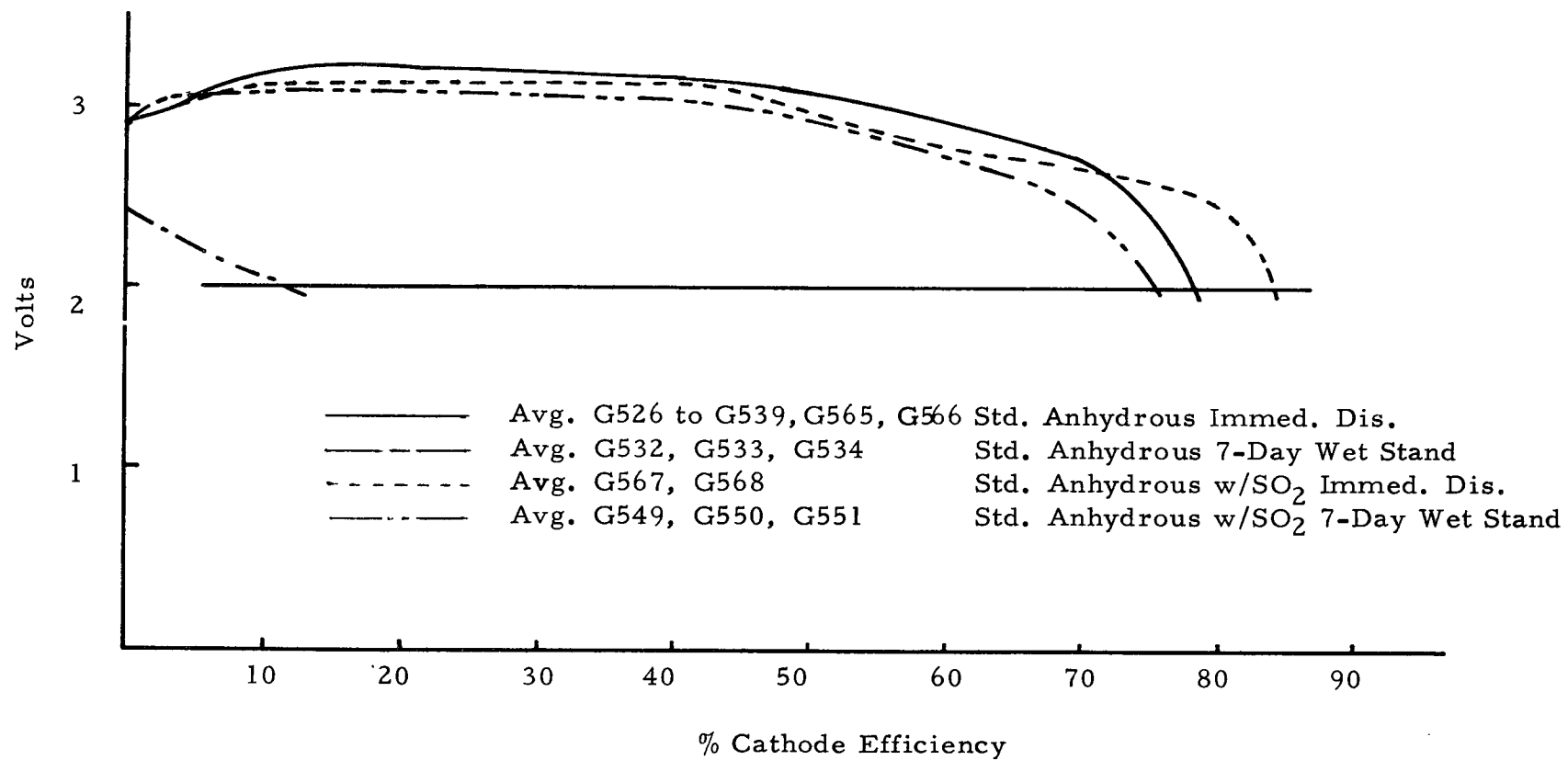


FORM FM-101

FIGURE 65

Discharge Performance of Cells in Experiment #18c

Experiment #25a  
+40°C  
10 ma/cm<sup>2</sup> at 3.0 volts

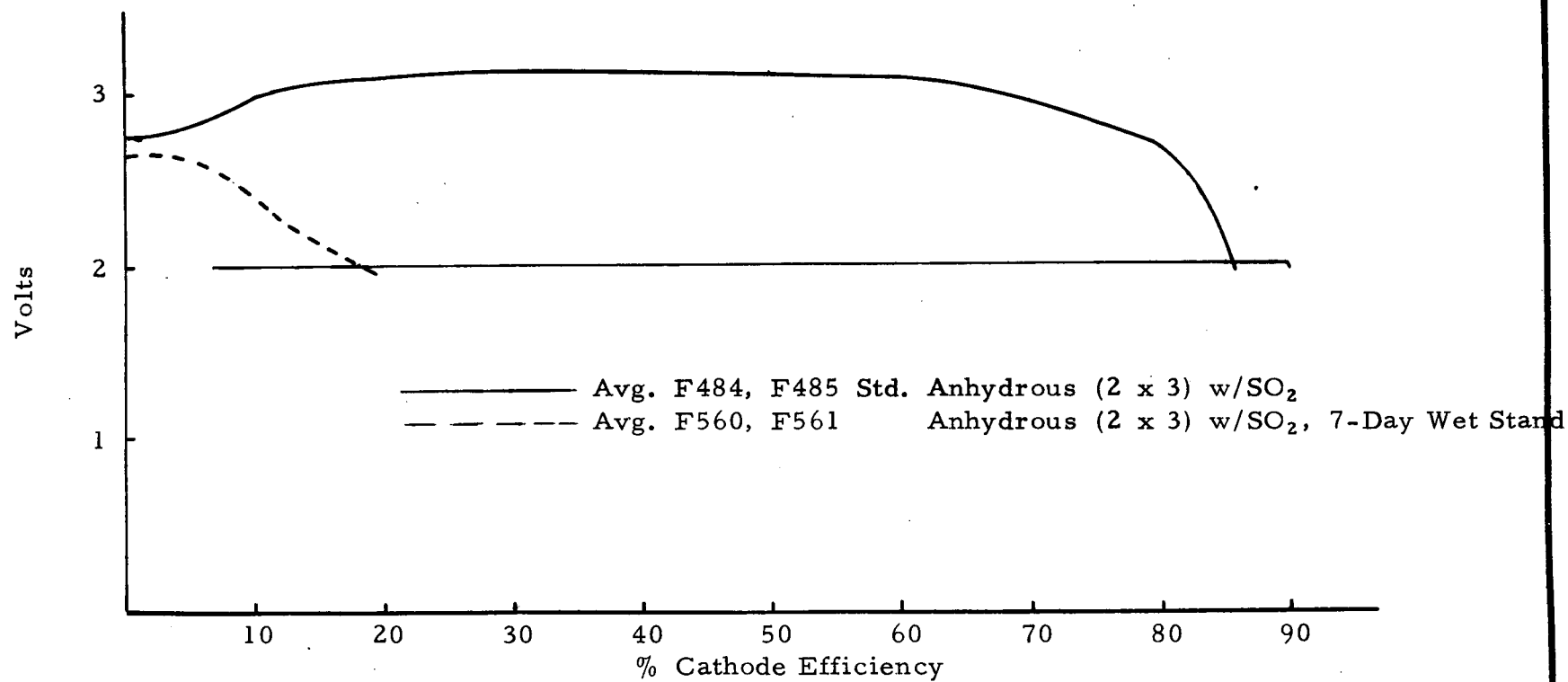


FORM FM-101

FIGURE 66

Discharge Performance of Cells in Experiment #25a

Experiment #25b  
+40°C  
10 ma/cm<sup>2</sup> at 3.0 volts



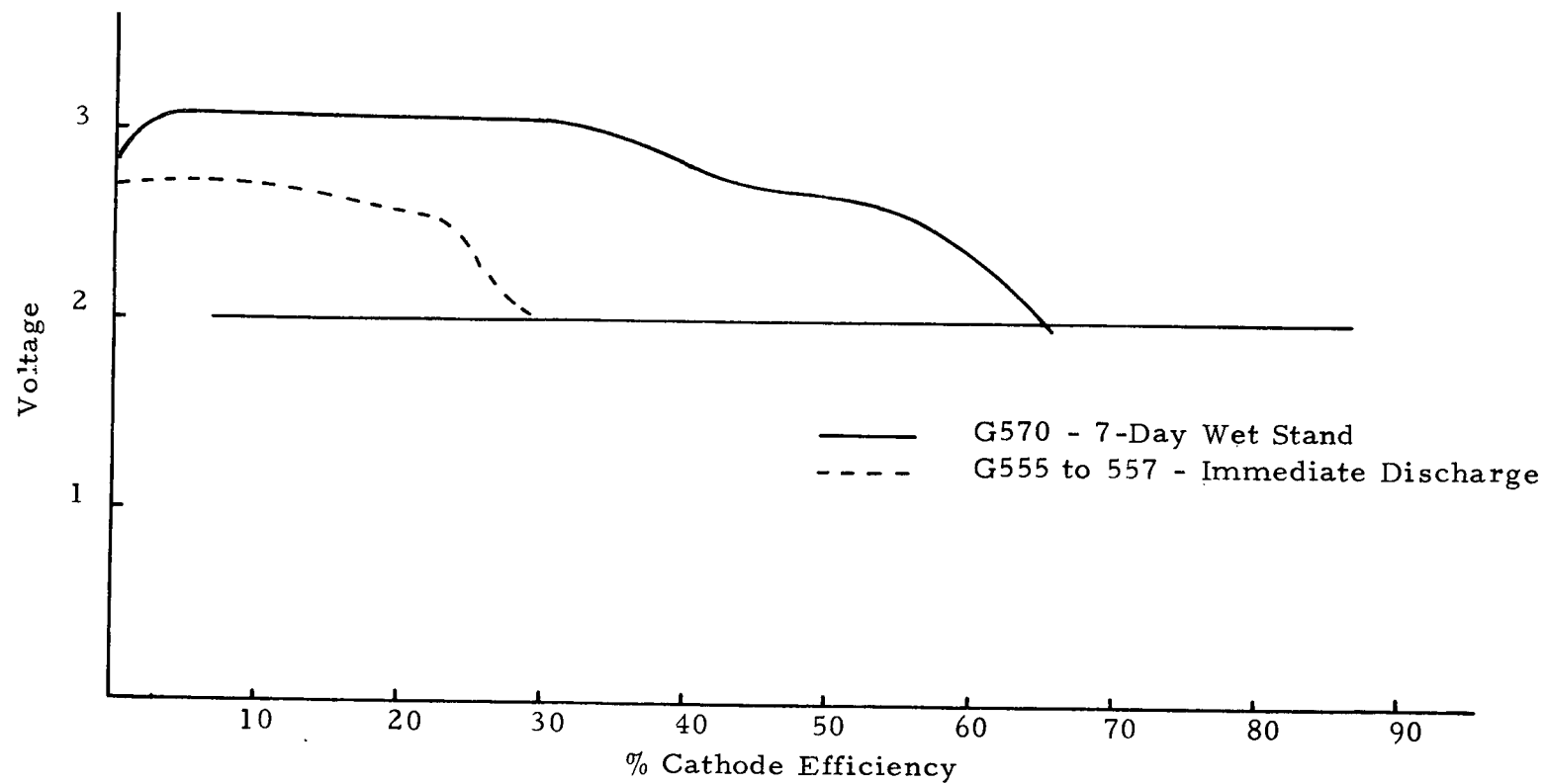
FORM FM-101

FIGURE 67

Discharge Performance of Cells in Experiment #25b

Experiment #25c  
+40°C  
10 ma/cm<sup>2</sup> at 3.0 v

Effect of Porvic/Glass Separator



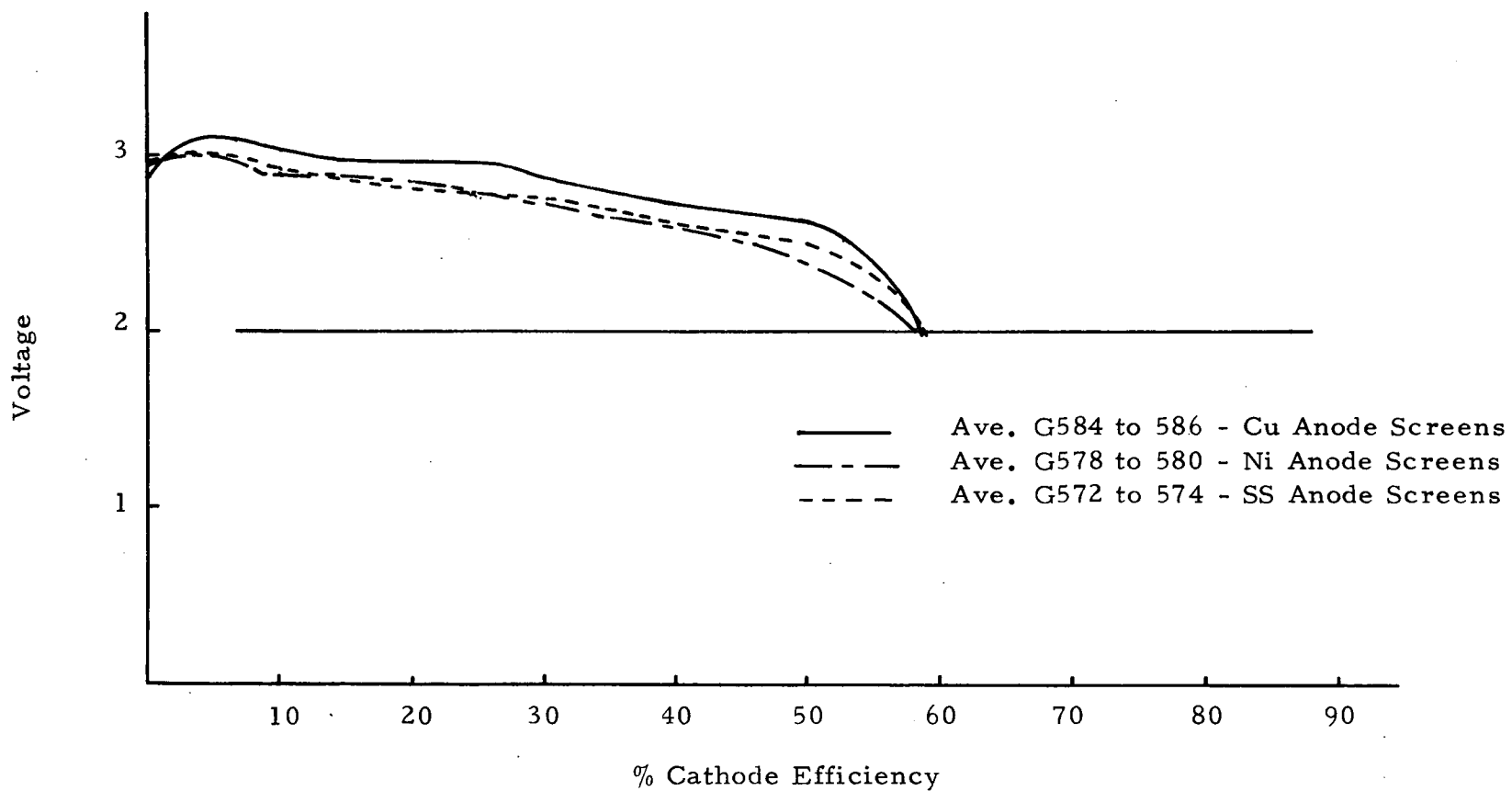
FORM FM-101

FIGURE 68

Discharge Performance of Cells in Experiment #25c

Experiment #27  
+40°C  
10 ma/cm<sup>2</sup> at 3.0 v

Seven-Day Active Stand - SO<sub>2</sub> Atmosphere



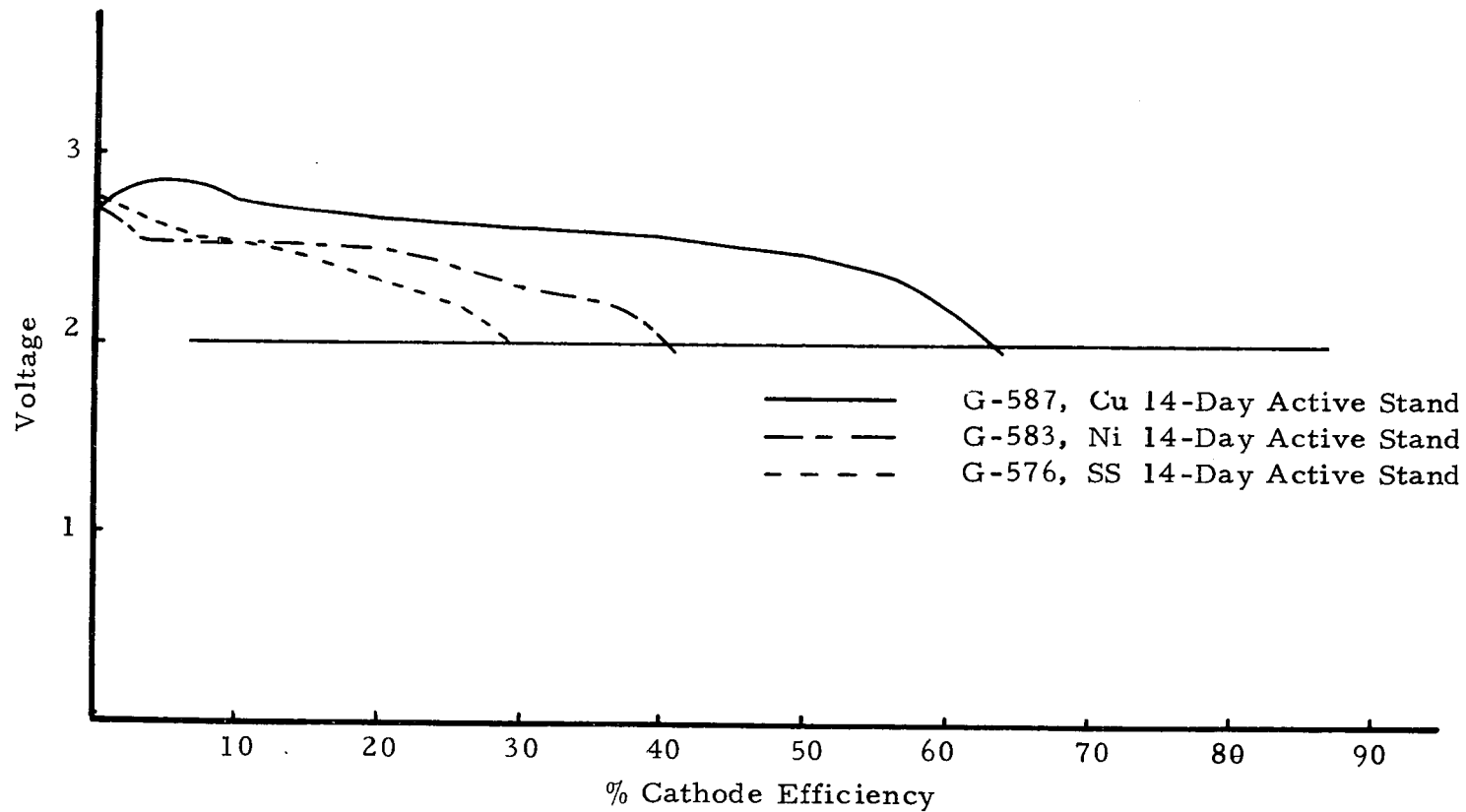
FORM FM-101

FIGURE 69

Discharge Performance of Cells in Experiment #27

Experiment #27  
5/7/71  
10 ma/cm<sup>2</sup> at 3.0 v  
+40°C

Effect of Anode Grid Material



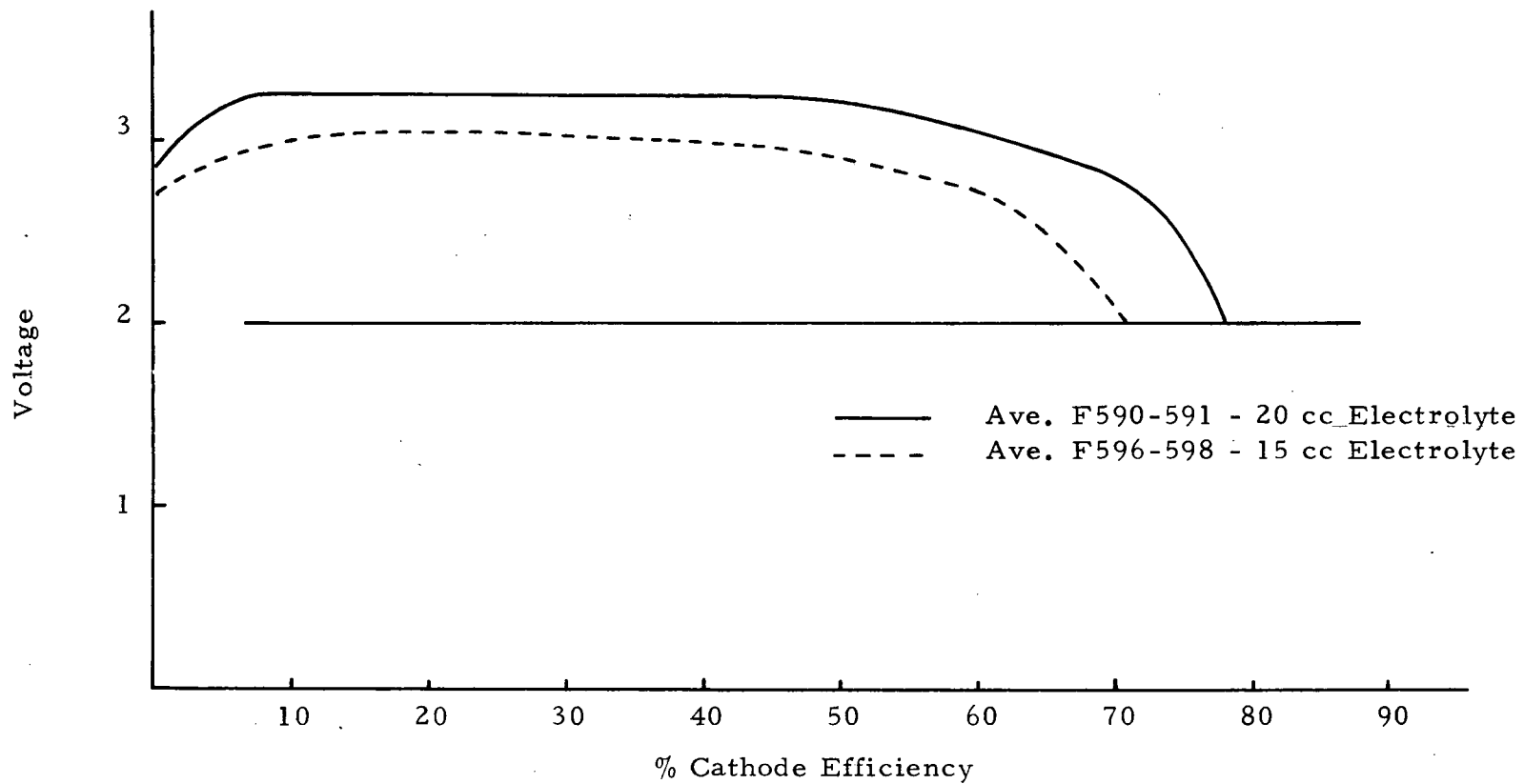
FORM FM-101

FIGURE 70

Discharge Performance of Cells in Experiment #27

Experiment #28  
+40°C  
10 ma/cm<sup>2</sup> at 3.0 v

### Effect of Electrolyte Volume



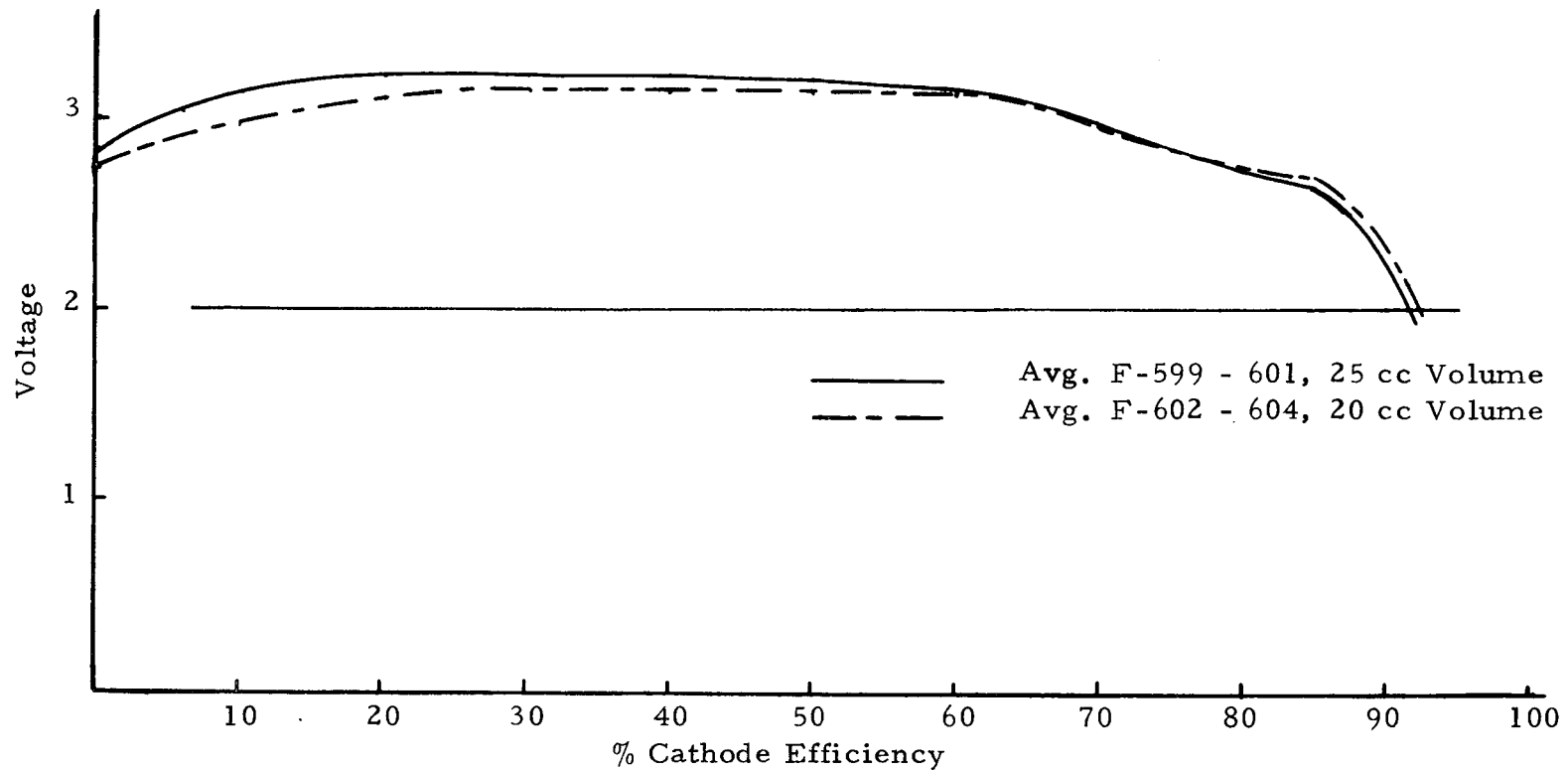
FORM FM-101

FIGURE 71

Discharge Performance of Cells in Experiment #28

Experiment #29  
5/12/71  
10 ma/cm<sup>2</sup> at 3.0 v  
+40°C

Effect of Electrolyte Volume, Immediate Discharge



FORM FM-101

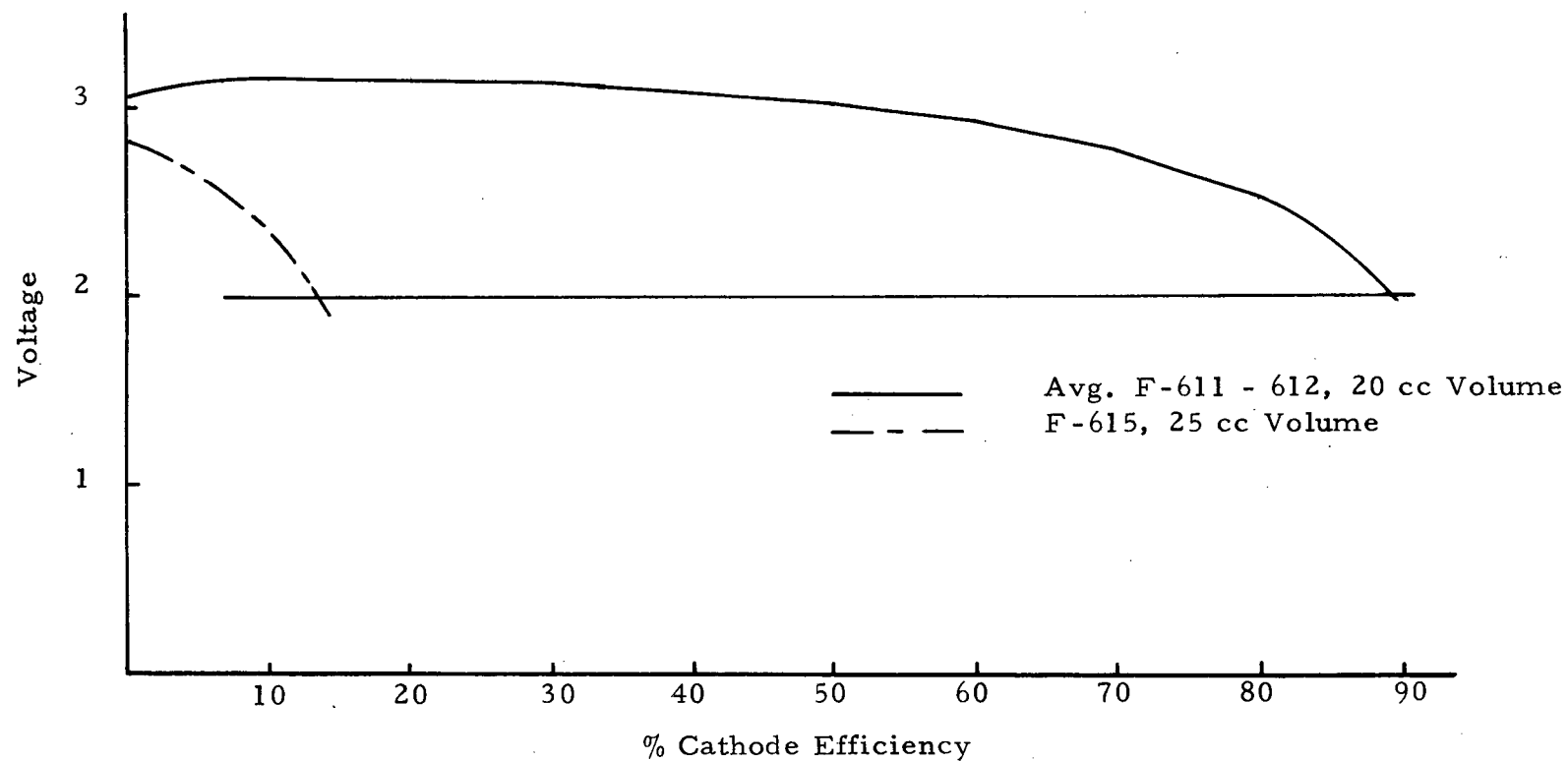
FIGURE 72

Discharge Performance of Cells in Experiment #29



Experiment #29  
5/26/71  
10 ma/cm<sup>2</sup> at 3.0 v  
+40°C

Effect of Electrolyte Volume, 14-Day Active Stand



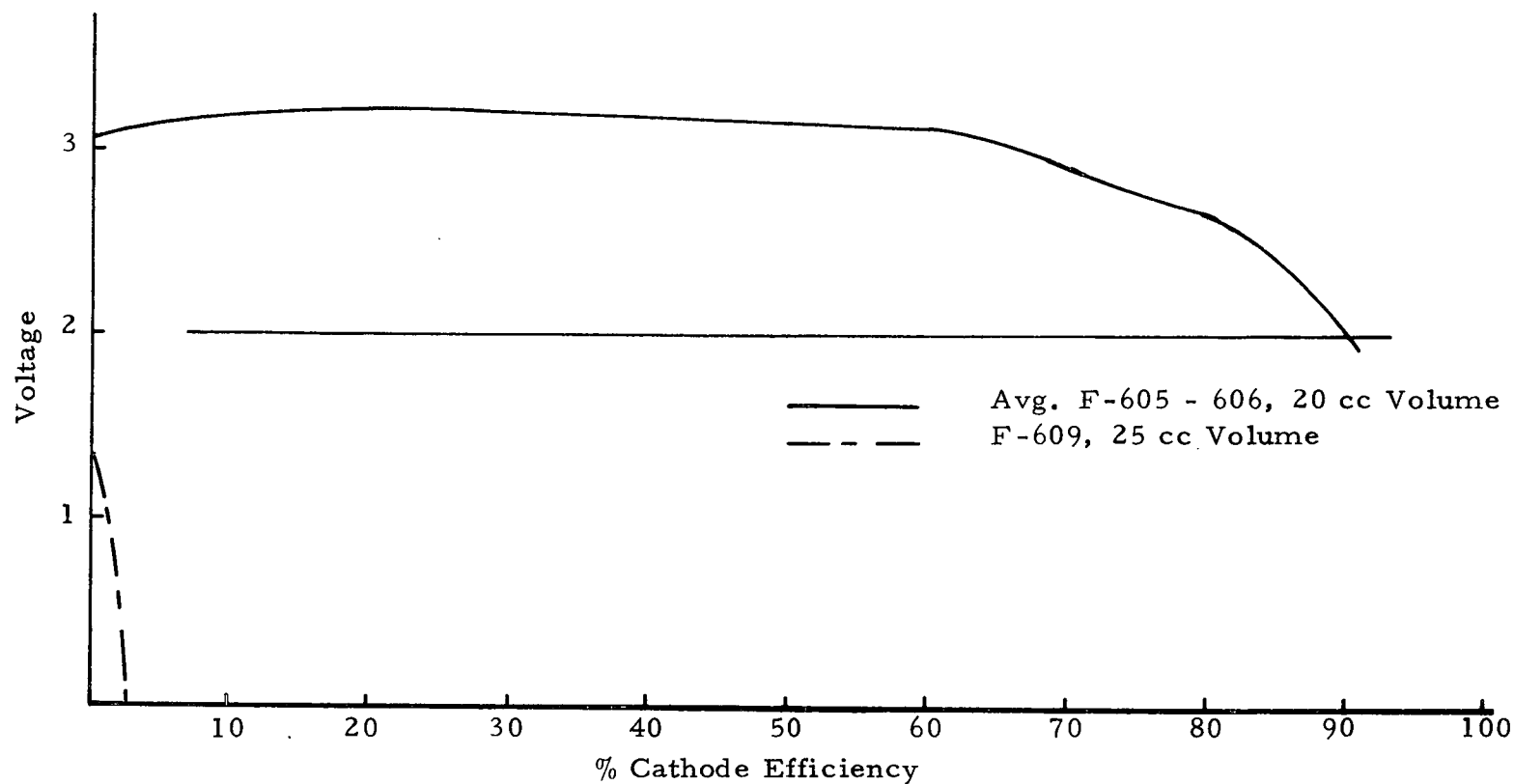
FORM FM-101

FIGURE 73

Discharge Performance of Cells in Experiment #29

Experiment #29  
5/19/71  
10 ma/cm<sup>2</sup> at 3.0 v  
+40°C

Effect of Electrolyte Volume, 7-Day Active Stand



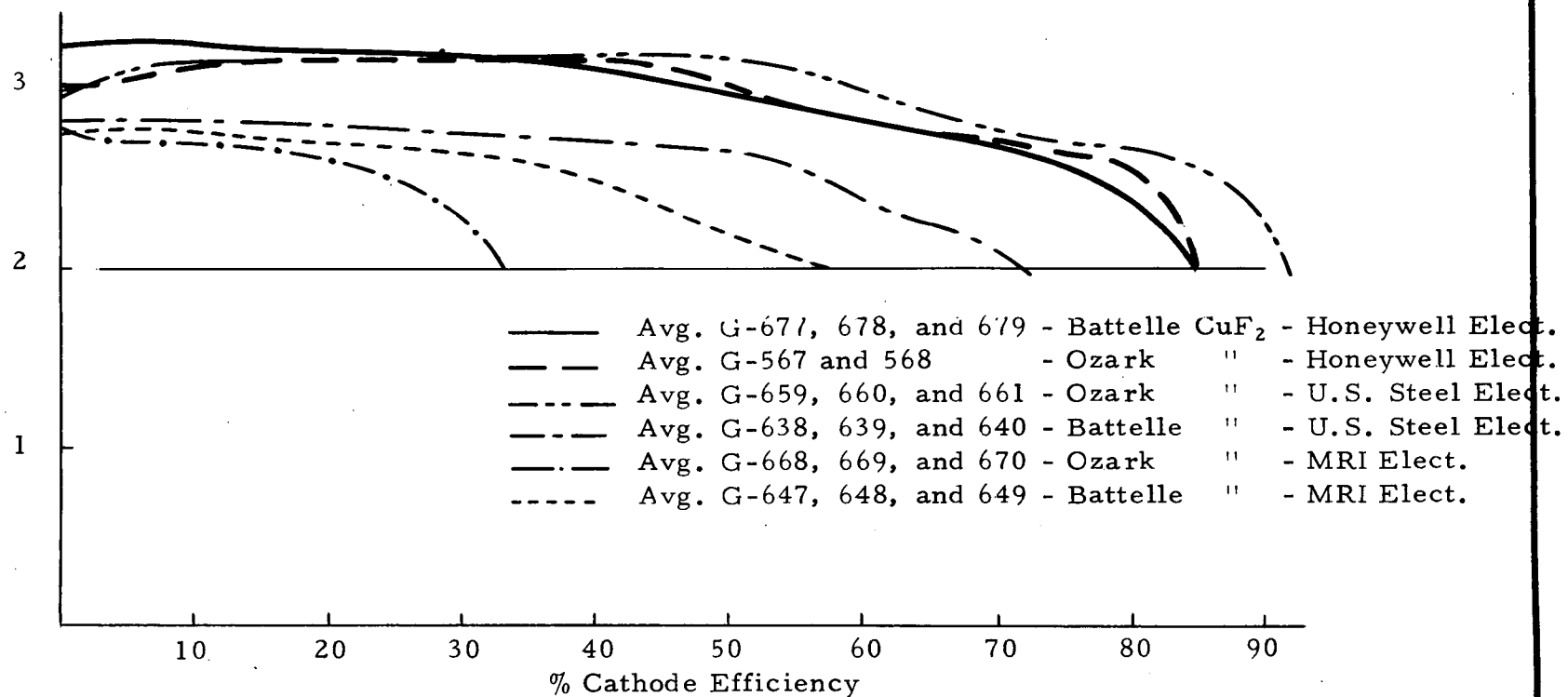
FORM FM-101

FIGURE 74

Discharge Performance of Cells in Experiment #29

Exp. 31  
+40°C  
10 ma/cm<sup>2</sup> at 3.0 v

# Effect of CuF<sub>2</sub> and Electrolyte on Immediate Discharge



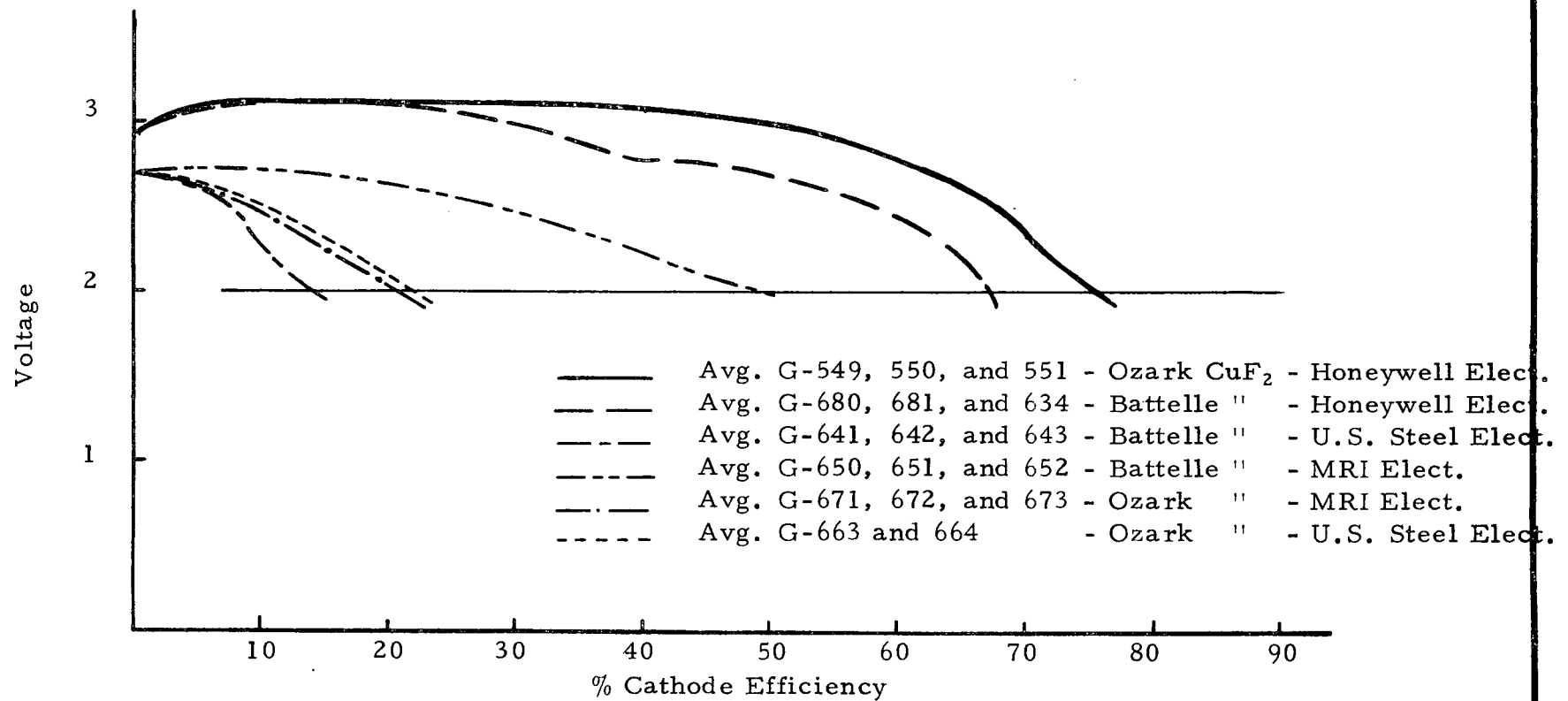
FORM FM-101

FIGURE 75

Discharge Performance of Cells in Experiment #31

Exp. 31  
+40°C  
10 ma/cm<sup>2</sup> at 3.0 v

# Effect of CuF<sub>2</sub> and Electrolyte After 7-Day Storage



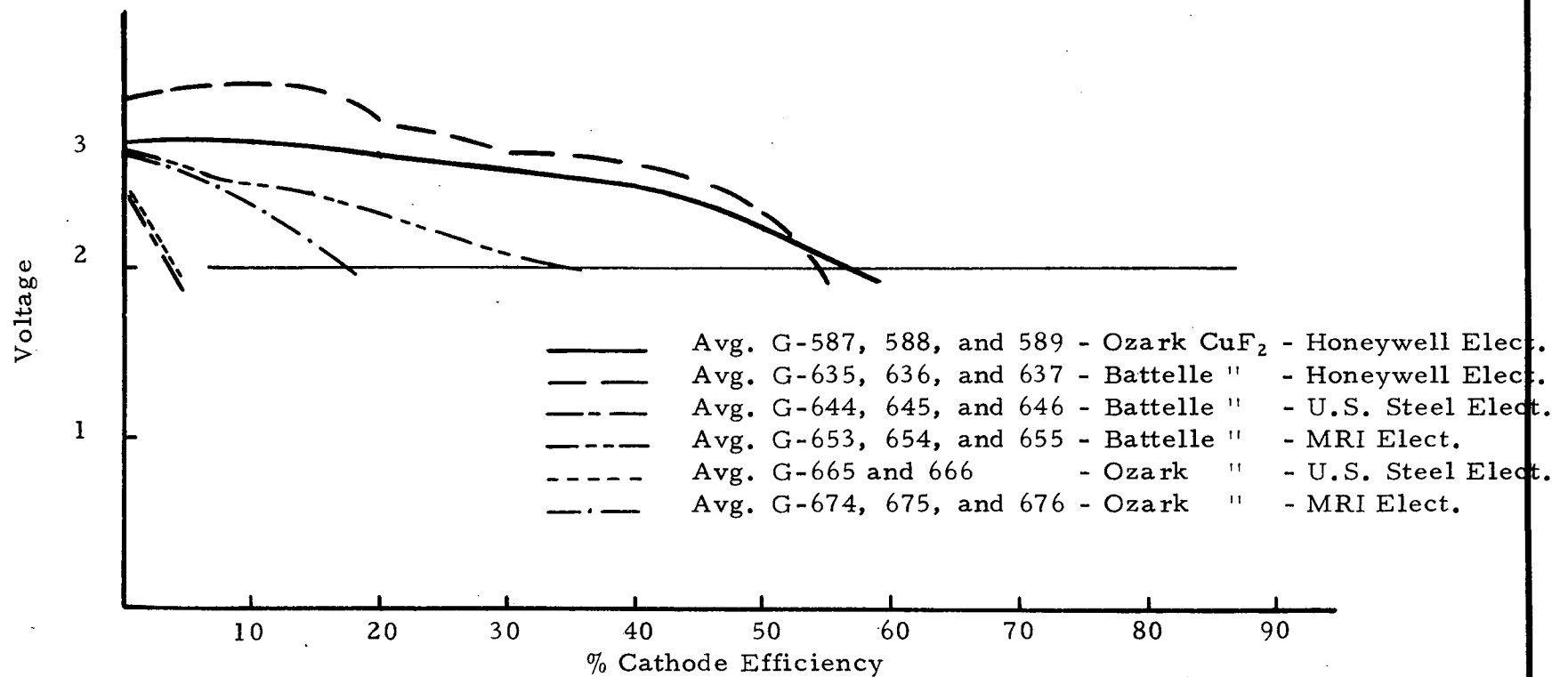
FORM FM-101

FIGURE 76

Discharge Performance of Cells in Experiment #31

Exp. 31  
+40°C  
10 ma/cm<sup>2</sup> at 3.0 v

# Effect of CuF<sub>2</sub> and Electrolyte After 14-Day Storage



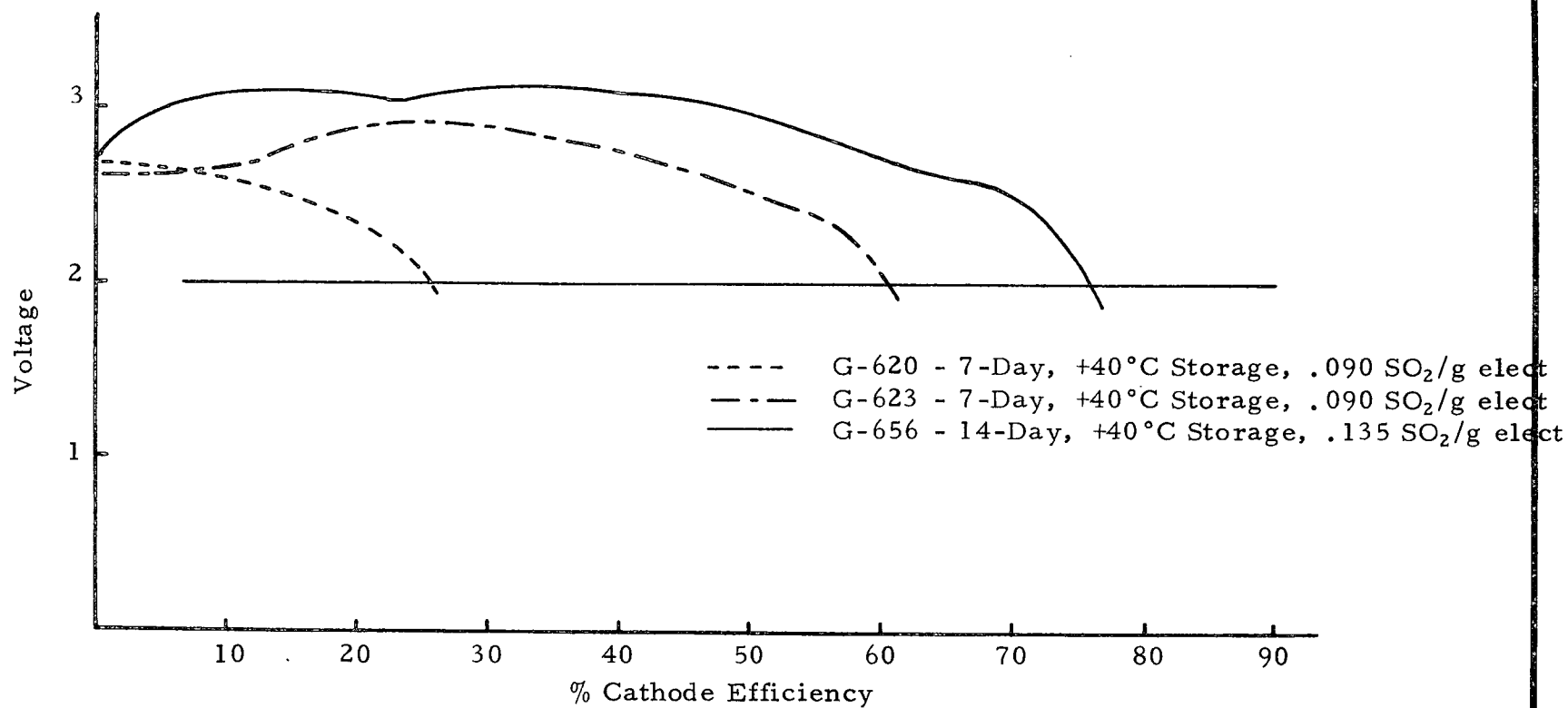
FORM FM-101

FIGURE 77

Discharge Performance of Cells in Experiment #31

Experiment #30  
5/28/71  
10 ma/cm<sup>2</sup> at 3.0 v  
+40°C

Effect of Low SO<sub>2</sub> on 7-14 Day Active Stand

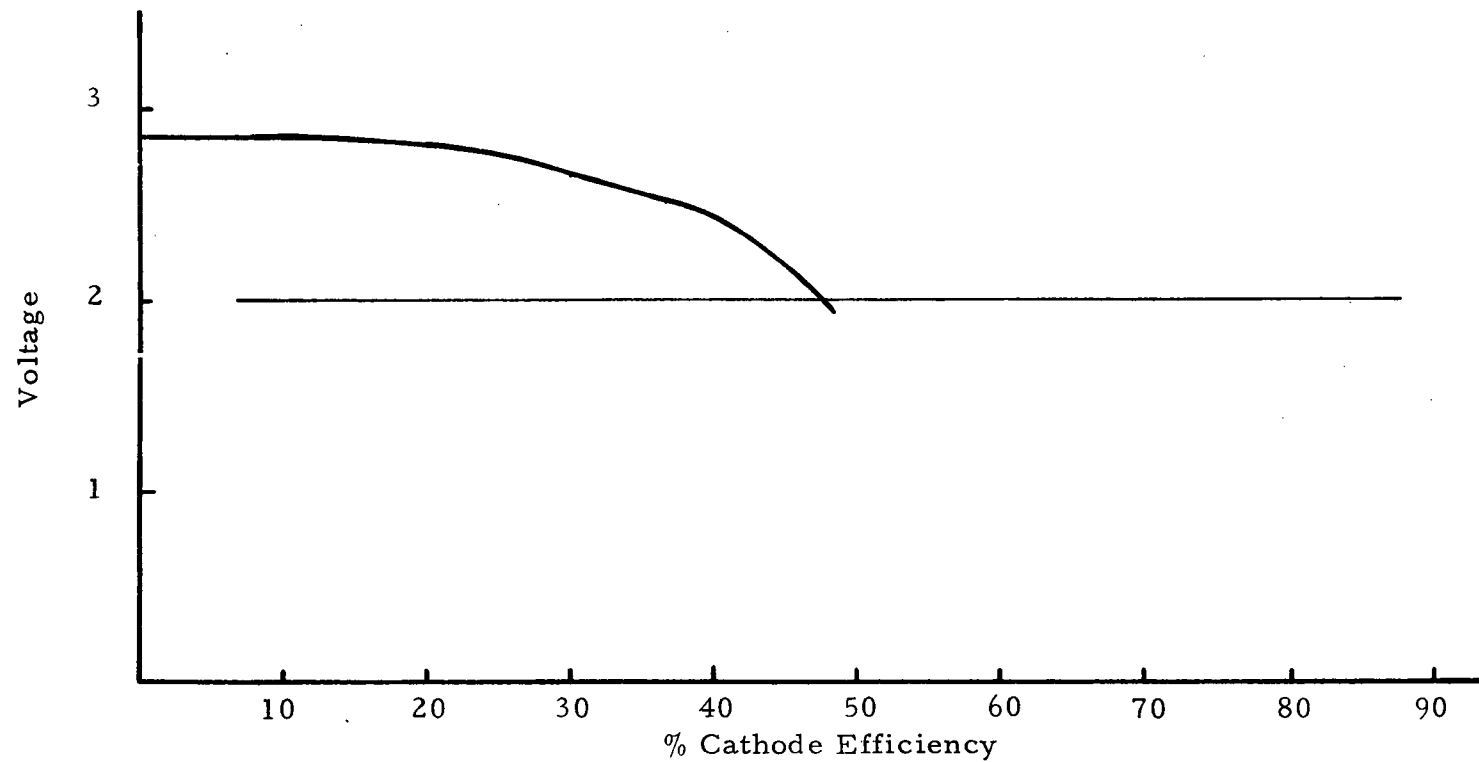


FORM FM-101

FIGURE 78

Discharge Performance of Cells in Experiment #30

Exp. 33  
Cell G-694  
+40°C  
7/2/71  
10 ma/cm<sup>2</sup> at 3.0 v  
.090 g SO<sub>2</sub>/g of Electrolyte



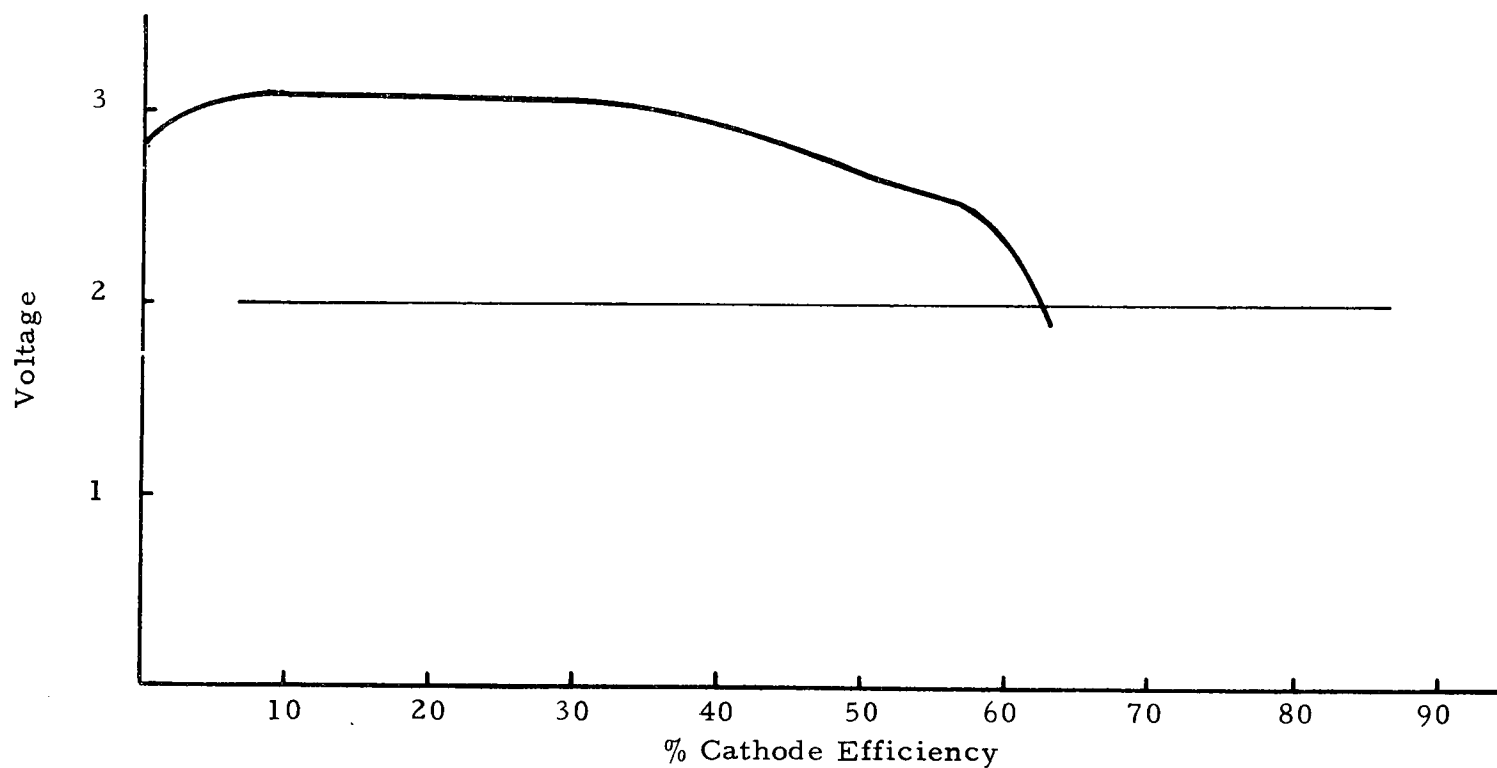
FORM FM-101

FIGURE 79

Discharge Performance of Cells in Experiment #33

Exp. 32  
Cell G-682  
+40°C  
7/2/71  
10 ma/cm<sup>2</sup> at 3.0 v

Electrolyte Age - 115 Days

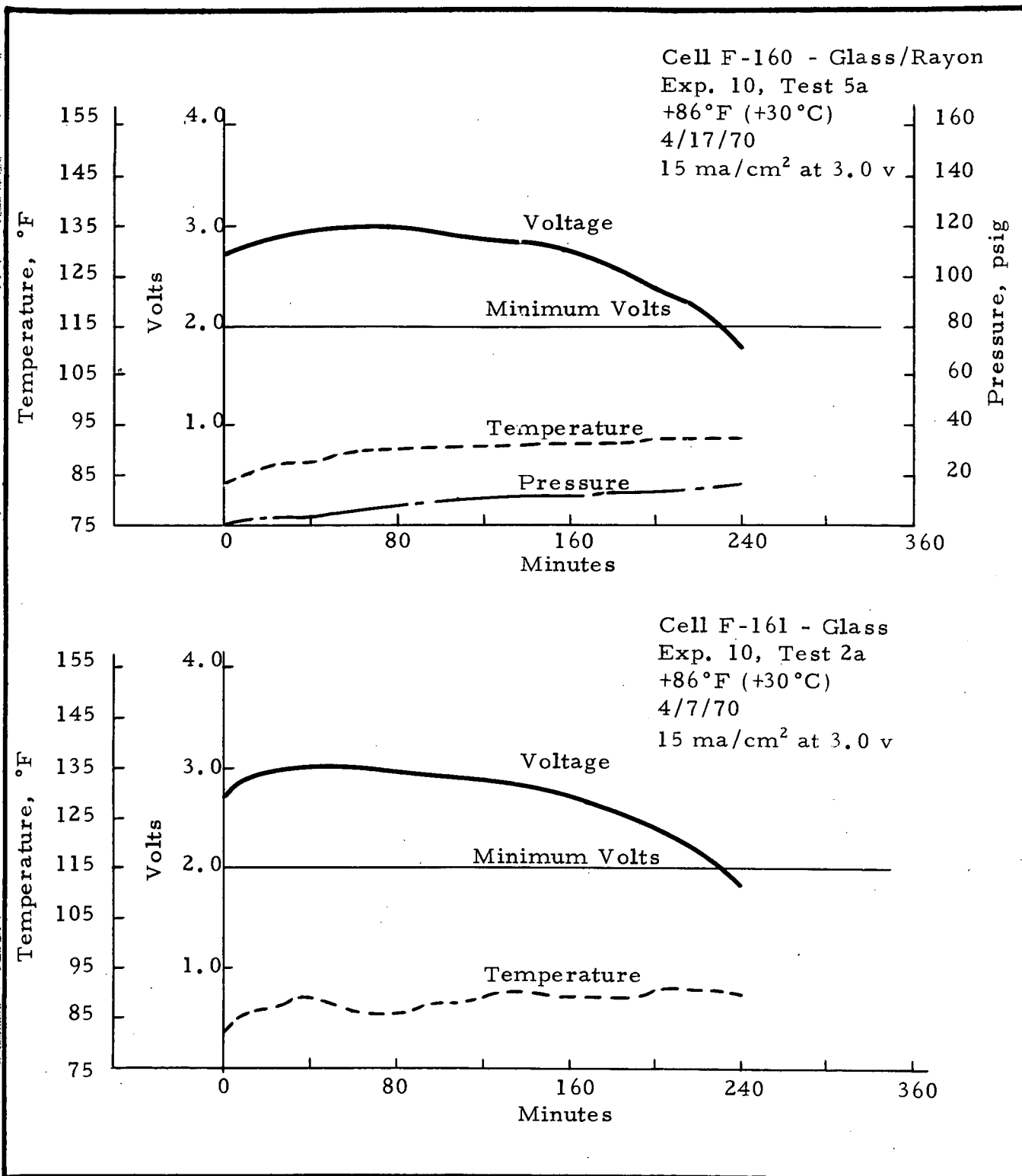


FORM FM-101

FIGURE 80

Discharge Performance of Cells in Experiment #32





FORM FM-100

FIGURE 81

Discharge Performance of Cells in Experiment #10

## APPENDIX "A"

Electrolyte Preparation  
Procedure PSC 8015-1

The following procedure is to be used in preparing the  $\text{LiAsF}_6/\text{MF}$  electrolyte necessary for the three tasks outlined in NASA Contract No. NAS 3-13221.

## 8015-1-A

Distillation of MF:

Step 1 - Bottles of stock MF required are removed from storage locker, event logged on MF control log (F-1). These bottles wear a blue label. The bottle numbers are logged in the proper column on MF distillation log (F-5). If a batch of previously distilled, but rejected, MF is used, the "date used" and "used for" columns are filled in the stock "bottle number" column for the new batch being run.

Step 2 - Preparation of Distillation Apparatus.

The apparatus is arranged according to Figure A-1.

Place the whole distillation system, including molecular sieve, under a hard vacuum for a minimum of 24 hours.

Air is replaced slowly through a drying tube containing a 4A sieve.

Step 3 - Distillation Process.

Remove stopper on top of 4A sieve column and install a 500 cc separatory funnel.

Introduce 400 cc of stock MF into the separatory funnel and adjust the flow rate to 60 cc/min. Continue adding the MF until 1400 cc have been collected in the distilling flask.

Remove 640 mm column and replace with funnel.

Quickly add the stirring bar and 1.4 grams of Li powder. Seal system with the glass stopper and stir rapidly for 1 hour.

Heat solution to a boil ( $31.5^\circ\text{C}$ ) and distill at a flow rate of 4.5 ml/min. Collect distillate in argon filled bottles vented to air through a drying tube packed with 4A sieve.

Discard the first and last 200 cc.

Step 4 - The distilled MF is checked by Karl Fischer analysis for water content according to procedure 8015-2, and then a GC is run to determine the amounts of MeOH, H<sub>2</sub>O, formic acid and other impurities greater than 200 ppm. The figures obtained will be recorded on the MF distillation log (F-5).

8015-1-B

# Recrystallization of $\text{KAsF}_6$

The salt is purified from its raw stock condition by recrystallization from water.

- Step 1 - Dissolve 720 grams of  $\text{KAsF}_6$  in 2000 ml of distilled water and heat to  $+60^\circ\text{C}$  in a 3000 ml beaker.
- Step 2 - Filter the solution through an 11 cm Buchner porcelain funnel using #42 Whatman filter paper into a 2-liter vacuum flask. Transfer the filtrate into a 3000 ml beaker.
- Step 3 - The filtrate is heated to  $+100^\circ\text{C}$  and evaporated to one-half its original volume.
- Step 4 - The solution is then allowed to recrystallize at room temperature ( $28^\circ\text{C}$ ) for a minimum of 12 hours. The crystals are then separated from the solution via suction filtration through an 11 cm Buchner funnel using #42 Whatman filter paper.
- Step 5 - Place the crystals in evaporating dishes (as large as possible bed area is preferred) and apply a soft vacuum (Gast air/vacuum pump) for 1 hour. Place the evaporating dish in a desiccator over concentrated  $\text{H}_2\text{SO}_4$  and apply a hard vacuum for 48 hours.
- Step 6 - At the end of step 5, a small sample of the large lot is placed in a drying bottle to a depth equivalent to that of the larger sample's bed depth. This sample gets the same drying treatment as the larger and is used for constant weight determination. At this point the salt and small sample are dried in a vacuum oven at  $110^\circ\text{C}$  for 48 hours. At the end of this period the small sample is weighed and a water determination made on the large sample. If the water level is not below 300 ppm, the salt is returned to the oven for another 24 hours at the end of which a new weight and water analysis is taken. This is continued until the water level is satisfactory.
- Step 7 - To determine  $\text{H}_2\text{O}$  content in the salt, dissolve a 1-gram sample into 10 ml of distilled MF. Titrate 1-cc samples of this by Karl Fischer. By knowing the initial water level of the MF and the water level after the salt is dissolved, the water in the salt can be calculated.

All necessary information is recorded on  $\text{KAsF}_6$  processing log (F-7). When the salt has an acceptable water level, it is packaged in Mason jars and stored in the process section of the NASA locker.

8015-1-C

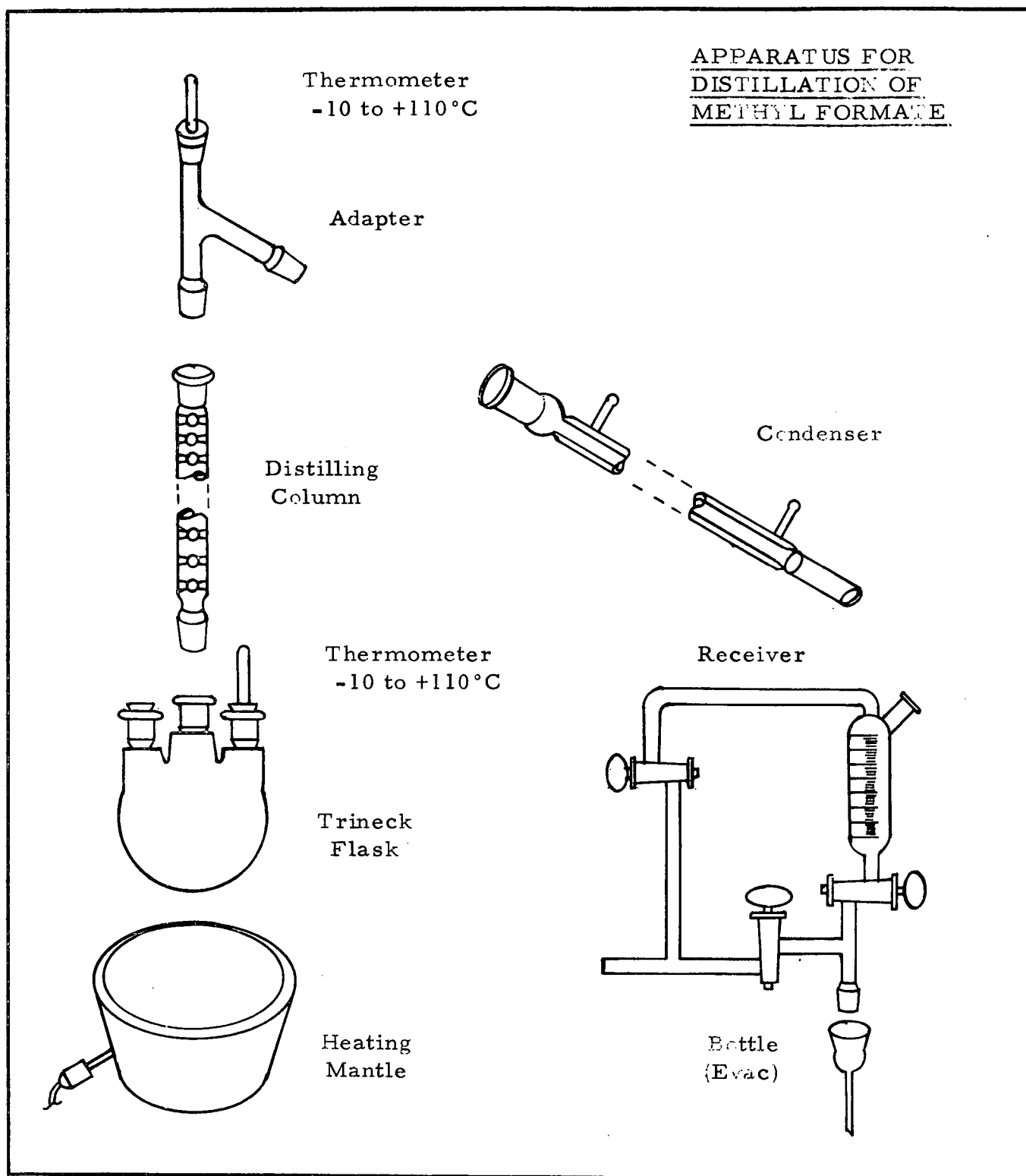
Drying of  $\text{LiBF}_4$

- Step 1 - The required weight of  $\text{LiBF}_4$  is removed from a stock bottle and placed in a shallow evaporating dish. The necessary notation is made on  $\text{LiBF}_4$  control log (F-2).
- Step 2 - The salt is dried for 48 hours in a vacuum oven at  $110^\circ\text{C}$ . A small sample in a weighing bottle of equivalent bed depth is dried along with the main sample.
- Step 3 - At the end of 48 hours, a weight is determined on the small sample, and a water analysis by KF is done on the large sample. The results are logged in the  $\text{LiBF}_4$  processing log (F-6).

If the water level is not below 500 ppm, the salt is recycled for 24 hours of vacuum/heat drying. At the end of the recycle, a new weight and water analysis is taken. If the number of cycles reaches five, consult engineer in charge.

The method for water analysis is as given under  $\text{KAsF}_6$  processing procedure 8015-1-B.

When the water level is acceptable, the salt is packaged in Mason jars (done in glove box) and stored in the NASA locker.



FORM FM-100

FIGURE A-1

Electrolyte Preparation  
Procedure PSC 8015-1 (Continued)

The following steps are used to make  $\text{LiAsF}_6/\text{MF}$  electrolyte. All operations where the materials are not in closed containers are performed in glove box #1 (Electrolyte Glove Box) which contains an argon atmosphere ( $\text{O}_2$  level  $< 1\%$ ).

- Step 1 - The required materials ( $\text{KAsF}_6$ ,  $\text{LiBF}_4$ , MF) are removed from the processed section of the NASA storage locker. The date removed and the purpose for removal are logged onto the respective sheets that apply (MF - F-5,  $\text{LiBF}_4$  F-6,  $\text{KAsF}_6$  F-7).
- Step 2 - The batch numbers of the constituents are logged onto sheet #1 of the electrolyte worksheet (F-9).

The water levels of each of the constituents in ppm's are logged onto the electrolyte data summary (F-10).

If the water level on the constituents has not been determined for a period of time greater than two weeks, it must be reanalyzed for water before it can be used in the electrolyte.

The electrolyte worksheet (F-9) is used to log all calculations generated during the manufacturing process.

All glassware used in the process is to be baked at  $110^\circ\text{C}$  for eight hours in a vacuum oven before using. All plastic apparatus receives eight hours of hard vacuum before using. The following weights result in 500 ml of 3M electrolyte. These can be adjusted by standard chemical calculations to obtain electrolytes of different molarity or different volume.

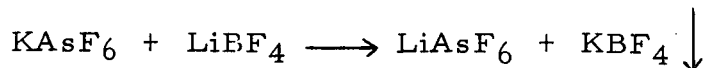
- Step 3 - Weigh out 342.03 grams  $\text{KAsF}_6$  in an appropriate glass container using a triple-beam scale.
- Step 4 - Weigh out 140.64 grams  $\text{LiBF}_4$  into a separate container.
- Step 5 - Dissolve the  $\text{KAsF}_6$  into 750 ml of distilled MF. Use a 2000-ml beaker and stir for one-half hour using a stirring bar and the magnetic stirrer fitted to the glove box.

CAUTION: Step 6 is exothermic in nature and care should be exercised to prevent excessive boiling from occurring.

- Step 6 - With the  $\text{KAsF}_6/\text{MF}$  solution stirring, slowly add the  $\text{LiBF}_4$  to the solution in small enough portions so as to minimize the boiling that

occurs. When the  $\text{LiBF}_4$  is completely added, stir the solution for a minimum of one hour.

The solution at this time contains a ppt of  $\text{KBF}_4$  as a result of the reaction:



Step 7 - Separate the precipitate from the solution via suction filtration through an 11 cm Buchner funnel using #42 Whatman paper (filter paper is dried 48 hours in a vacuum oven). If method A is to be followed, the ppt is washed with 3, 50-ml portions of distilled MF. If method B is used, only 1, 50-ml portion of MF is used.

Steps 8, 9 and 10 can be done by two methods. It is necessary to check with the engineer in charge at this time to establish the method to be used.

- A. Stoichiometric Comparison
- B. Specific Gravity Comparison

If method A is to be used, proceed through steps 8A, 9A and 10A; if method B is to be used, proceed to steps 8B, 9B and 10B. The procedure becomes the same for both beginning with Step 11.

#### Method A - Stoichiometric Comparison:

Step 8A - Place the precipitate, filter and beaker, which was used to hold the solution prior to filtration, into a desiccator and evacuate using a Gast air pump for one-half hour to remove the major portion of residual MF. After the "soft" vacuum, place the desiccator under hard vacuum (30 inches Hg) for a minimum of eight hours.

Step 9A - Weigh the beaker and filter/precipitate combination, and from original tare weights determine the total weight of precipitate. The theoretical weight should be 188.8 grams for the example used in the procedure.

Step 10A - Check the determined precipitate weight against the theoretical. Agreement should be within  $\pm 2\%$  of the theoretical value ( $\pm 3.8$  grams for the example weights used in this procedure). If the deviation is greater than this, stop the process here and consult the engineer in charge before continuing.

If the deviation is within range and:



- (a) in excess of theoretical, the excess is considered to be  $\text{LiAsF}_6/\text{MF}$  not in solution but salted out in the precipitate.
- (b) below theoretical, this is considered indicative of incomplete reaction.

Depending on whether (a) or (b) applies, the appropriate calculations is made to determine the  $\text{LiAsF}_6$  in solution and the volume necessary to give the desired final molarity. This volume is 500 ml for the example weights given in the procedure.

Method B - Specific Gravity Comparison:

Step 8B - Determine the specific gravity of the filtrate using a calibrated 10-cc pycnometer. Sample size is two.

Step 9B - Measure the volume of the remaining solution using the 1800-ml vacuum flask.

Step 10B - With the values obtained in steps 8 and 9, determine the molarity using the specific gravity vs. molarity chart (C-1), the number of moles of  $\text{LiAsF}_6$  in solution and the volume of solution necessary to obtain the desired molarity. This volume is 500 ml for the weights given in the procedure. The precipitate is discarded at this point; there is no need to vacuum dry or check the weight.

Step 11 - The desired volume is obtained by one of the following:

- (a) Add distilled MF to dilute to the proper level.
- (b) Apply a vacuum to the graduated vacuum bottle to evaporate MF until the required volume is obtained.

For either (a) or (b) an 1800-ml graduated vacuum flask is used to contain and measure the liquid.

Step 12 - The adjusted solution is bottled as follows:

- (a) A 50 to 100-ml sample is placed in a 100-cc bottle with a strip of argon packed lithium--the bottle is sealed with a rubber stopper which is then sealed with plastic adhesive tape. The bottle is labeled and stored.
- (b) The balance of solution is packaged in a 500 or 1000-ml bottle whichever is appropriate, also with a strip of lithium and is sealed in the same manner as the small sample. The bottle is labeled as to its batch #, contents, molarity, and date of manufacture.

Step 13 - The solution is then submitted to the following quality control checks.

- A. Water content by Karl Fischer analysis (procedure 8015-2).
- B. Specific Conductivity
- C. Specific Gravity

These data along with processing figures are recorded on the electrolyte worksheet (F-9), and the required data are recorded on form F-10 electrolyte data summary.

NAS 3-13221

F-9a

Electrolyte Worksheet

Batch No. MF \_\_\_\_\_  
 Batch No. KAsF<sub>6</sub> \_\_\_\_\_  
 Batch No. LiBF<sub>4</sub> \_\_\_\_\_

Electrolyte Batch No. NASA -  
 Date Start Mfd. \_\_\_\_\_  
 Molarity \_\_\_\_\_

Mole Weight KAsF<sub>6</sub> - 228.02  
 Mole Weight LiBF<sub>4</sub> - 93.76  
 Mole Weight LiAsF<sub>6</sub>·MF - 255.91  
 Mole Weight KBF<sub>4</sub> - 125.92

Grams KAsF<sub>6</sub> \_\_\_\_\_ = Moles LiBF<sub>4</sub> x 93.76 x 1.02 = \_\_\_\_\_  
 Moles KAsF<sub>6</sub> \_\_\_\_\_ g LiBF<sub>4</sub> req.

Method A:

(1) Gross funnel + beaker and paper + ppt	_____	Funnel Tare	_____
(2) Tare funnel + beaker + paper	_____	Beaker Tare	_____
(3) Weight KBF <sub>4</sub> ppt	_____	Paper Tare	_____
(4) Theory - ppt weight (moles KAsF <sub>6</sub> x 125.92)	_____		
(5) Weight difference (3-4)	_____		
(6) Percent error (5) ÷ (4)	_____	(Limit ± 2%)	
(7) Moles LiAsF <sub>6</sub> lost:			
If (5) is + than (5) ÷ 255.91	_____		
If (5) is - than (5) ÷ 125.92	_____		
(8) Moles LiAsF <sub>6</sub> in solution [moles KAsF <sub>6</sub> - (7)]	_____		
(9) Required volume [(8) ÷ molarity]	_____	liters	

Method B:

Specific gravity filtrate      Sample No. 1 \_\_\_\_\_  
    Sample No. 2 \_\_\_\_\_

(1) Average of Sample Nos. 1 and 2	_____	
(2) Volume filtrate	_____	liters
(3) Molarity filtrate using Chart 1 and value (1)	_____	
(4) Moles LiAsF <sub>6</sub> in solution (3) x (2)	_____	
(5) Required volume (4) ÷ molarity	_____	liters

NAS 3-13221

F-9b

Volume Adjustment

- (1) Required end volume \_\_\_\_\_  
 (2) Volume before adjustment \_\_\_\_\_  
 (3) Correction  $\Delta(1) - (2)$   $\pm$  \_\_\_\_\_  
 (4) Actual end volume \_\_\_\_\_  
 (5) Final molarity \_\_\_\_\_  
     Moles  $\text{LiAsF}_6 \div (4)$  \_\_\_\_\_

## Physical Checks:

Specific Gravity - Sample No. 1 \_\_\_\_\_  
                             Sample No. 2 \_\_\_\_\_  
                             Average \_\_\_\_\_

## Specific Conductivity -

Cell Constant \_\_\_\_\_ Date \_\_\_\_\_

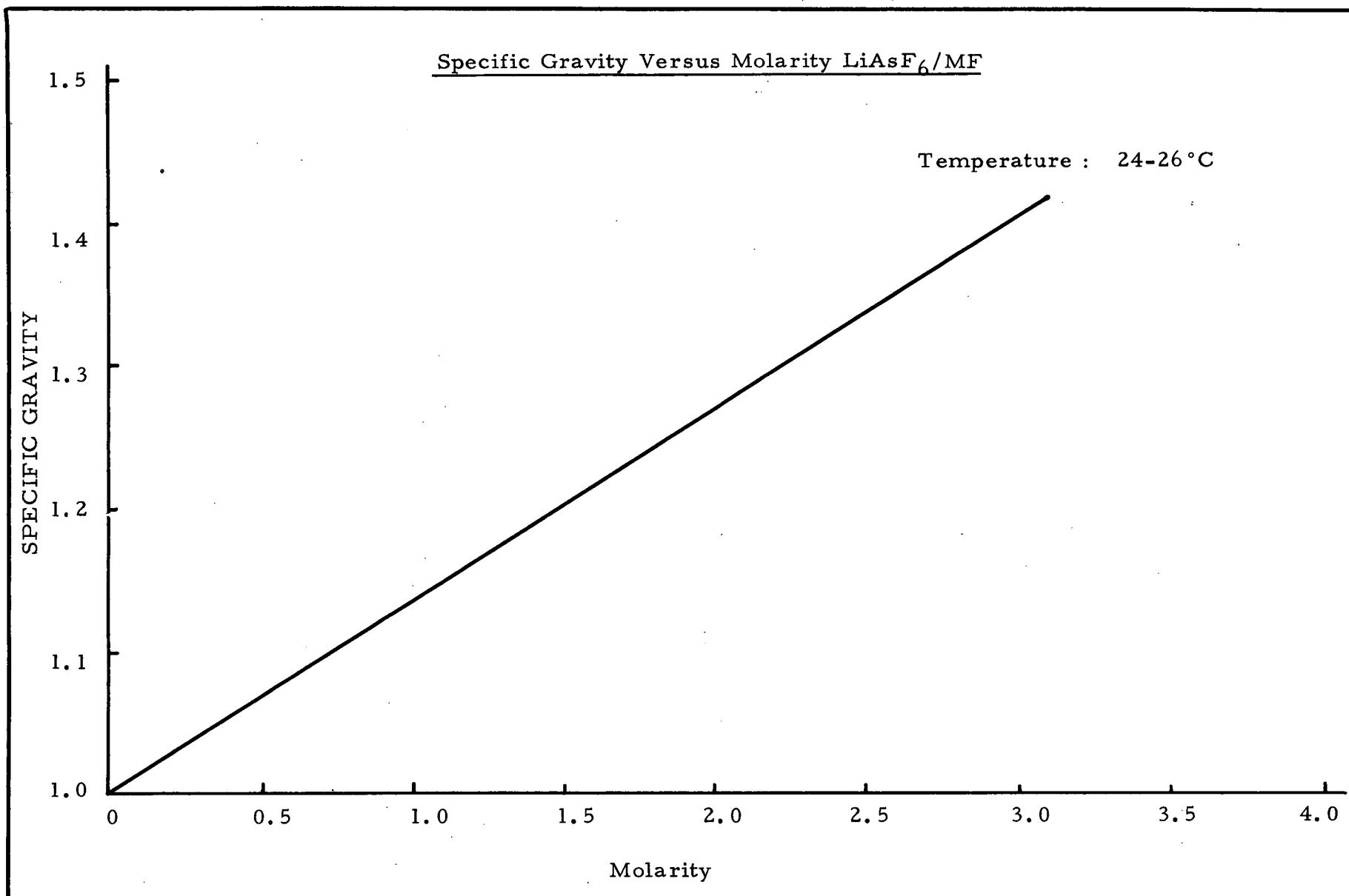
Temp. \_\_\_\_\_ Sample No. 1:  $\div$  \_\_\_\_\_ ohms x 1000 = \_\_\_\_\_  
                             (Cell Constant  $\text{cm}^{-1}$ )  
 Temp. \_\_\_\_\_ Sample No. 2:  $\div$  \_\_\_\_\_ ohms x 1000 = \_\_\_\_\_  
                             (Cell Constant  $\text{cm}^{-1}$ )  
 Temp. \_\_\_\_\_ Sample No. 3:  $\div$  \_\_\_\_\_ ohms x 1000 = \_\_\_\_\_  
                             (Cell Constant  $\text{cm}^{-1}$ )      Avg. \_\_\_\_\_ mmhos

Avg. \_\_\_\_\_

## Karl Fischer Water Analysis -

Titer Value \_\_\_\_\_ Date \_\_\_\_\_ Operator \_\_\_\_\_

Sample No. 1	_____ $\mu\text{l}$ KF x	_____ TV x	_____ Sp G =	_____ ppm
Sample No. 2	_____ $\mu\text{l}$ KF x	_____ TV x	_____ Sp G =	_____ ppm
Sample No. 3	_____ $\mu\text{l}$ KF x	_____ TV x	_____ Sp G =	_____ ppm
			Avg	_____ ppm



FORM FM-101

FIGURE A-2

## APPENDIX "B"

Karl Fischer Analysis  
Procedure PSC 8015-2

The following procedure will be used to determine the water content in the materials associated with the  $\text{LiAsF}_6$  electrolyte.

## Materials and apparatus required:

Karl Fischer Reagent  
Methanol Reagent Grade

- 2 - 1 cc syringe
- 3 - serum syringes
- 1 - 250  $\mu\text{l}$  syringe
- 1 - 25 cc syringe
- 3 - 10 ml vials
  
- 3 - Standard Needles
- 1 - #22G 1.5" Needle

All apparatus is maintained under a vacuum  $> 29"$  mercury prior to use. Vials are baked at  $+150^\circ\text{C}$  in a vacuum oven for 24 hours. A supply of these is maintained.

Procedure:

- Step 1 - Place serum stoppers in the three 10 ml vials. Using the 25 cc syringe pull a partial vacuum on each vial.
- Step 2 - Add one ml of methanol with the one cc syringe to each vial and shake vigorously for a few seconds.
- Step 3 - Add KF Reagent to each vial with the 250  $\mu\text{l}$  syringe fitted with the #22G 1.5" needle until the solution turns a light brown.
- Step 4 - Repeat Steps 3 and 4 for each vial.
- Step 5 - Add one ml of the sample to be tested to each vial.
- Step 6 - Add KF Reagent until the light brown end point occurs, and note the microliters of Reagent necessary to reach this point.
- Step 7 - To determine the  $\text{H}_2\text{O}$  content, multiply the number of microliters obtained for each vial in Step 6 by the titer value. The units will be mg of  $\text{H}_2\text{O}$ /ml solution. The value can be converted to ppm by dividing by the specific gravity of the solution.

Titer Value:

To obtain this value, follow the above procedure for Steps 1 through 4. Step 5 substitute, one ml of certified standard water to each vial. Then add the KF Reagent until the light brown end point occurs.

Divide the number of microliters of KF Reagent obtained into the value of the standard water which should be in mg H<sub>2</sub>O/ml solution. The quotient multiplied by 1000 is the titer value.

$$\frac{\text{Std. H}_2\text{O Value}}{\mu\text{l of KF}} \times 1000 = \text{Titer Value}$$

Notes:

- 1 Baking of bottles and storage in vacuum are essential.
- 2 When a titration is started, run to completion without interruption. Too long an interval between titrations (methanol, sample, etc.) will distort results.
- 3 The titer value should be determined for every day that samples are to be tested.
- 4 Each individual technician should determine his own titer value -- this will help minimize differences in individual end point determinations.
- 5 Diluted KF Reagent with a titer range of 1-2 is desirable for samples containing less than 200 ppm H<sub>2</sub>O. Other titrations can be done with titer values in 4-5 range which is value of "as received" material.

## APPENDIX "C"

Compatibility Study  
 Procedure PSC 8015-3

The following procedure will be used to determine the compatibility of candidate separator materials to 3 M LiAsF<sub>6</sub>/MF at 50°C (122°F) for 100 hours.

The following materials are under initial investigation:

<u>Materials</u>	<u>Manufacturer</u>
1) Glass fibre - filter matte, grade 934 AH	Reeve Angle
2) Pellon, polypropylene, FT 2140	Pellon
3) Porvic, synpor, microporous PVC	Povair, Ltd.
4) Weblox, 100% dacron EV 100	Kendall
5) Weblox, rayon EV 101	Kendall
6) Tyvec, 100% polyethylene 1458	DuPont

Pretest Procedure:

Step#1 -

- 1) Materials are inspected, classified and batch number logged.
- 2) Cut to appropriate size.
- 3) Weighed to 4 decimal places.
- 4) Measured for length and width to nearest .001".
- 5) Measured for thickness with sample compressed.
- 6) Measured for thickness with sample uncompressed.

Step #2

- 1) Samples are vacuumed for 24 hours at 30" of Hg (room ambient).



Test Procedure:

The following procedure will be used to perform Task I separator evaluation.

**Step #1 - Electrolyte Control**

- 1) Log batch number of electrolyte and manufacture date.
- 2) Determine water content by Karl Fisher analysis and record. (If the same batch is in use for a period of time, the H<sub>2</sub>O content is rechecked every 14 days.)
- 3) Log specific gravity.
- 4) Log specific conductivity.

**Step #2 - Preparation of Compatibility Samples**

- 1) Components of the compatibility fixture are thoroughly cleaned with distilled water, rinsed in acetone and hard vacuum for 24 hours.
- 2) Just prior to start of compatibility study perform the following:
  - a) Rinse tube with 10 cc of methyl formate.
  - b) Rinse tube with 5 cc of 3 M LiAsF<sub>6</sub>/MF.
- 3) Charge tube with 30 cc of 3 M LiAsF<sub>6</sub>/MF using a syringe for transport.
- 4) Insert separator sample into the tube with forceps.
  - a) Seal tube and add identification to the outside of tube.
- 5) After the above is completed for all samples, prepare a tube with just electrolyte as a control. This will go through the same treatment as those containing separator materials.
- 6) A sample of electrolyte is stored under ambient work area conditions to also serve as a control.
- 7) Place tubes into the 50°C environment. Monitor and record temperature using thermocouples.
  - a) After one hour check tubes to establish that each is showing pressure on gauge indicating a good seal.

- 8) If a leak is detected during the test cycle, that tube is removed and voided.

Step #3 - At 24-hour intervals, while on test, visually check the following and record:

- 1) Any change in color of the electrolyte by comparing to the control.
- 2) Level of vapor pressure, any leaks occurring; that test must be voided.
- 3) Changes in physical characteristics of the separator material.

Step #4 - Completion of Test Cycle (At the End of 100 Hours)

- 1) Remove test samples from environmental chamber and allow fixture to stabilize at room ambient before dismantling to prevent excessive liquid evaporation.
- 2) Dismantle test fixture.
- 3) Immediately take a 5 cc sample of electrolyte with a syringe. This will be used to check water content by Karl Fisher analysis (record).
- 4) Remove separator materials and note any physical changes.
- 5) Rinse separator materials in pure MF using the following method:
  - a) Material is rinsed sequentially in three (3) beakers containing 200-300 cc methyl formate for two (2) minutes each.
  - b) In the final rinse bath, monitor for salt level increase with a dip cell conductivity bridge.
  - c) When the conductivity in the third rinse increases by 5 millimhos over the pure MF reading, the MF is changed in all three (3) beakers.
- 6) After the third rinse, the separator samples are dried under a glass plate.
- 7) The samples are then vacuum dried at room ambient for 15 hours. Samples should be placed on an inert surface to prevent contamination.

Step #5 - Evaluation of Materials

- 1) Repeat initial measurements and record weight and size.
- 2) Calculate percent of change from original values.
- 3) Examine materials under a microscope next to an untreated sample and identify any differences.
- 4) Determine relative tear strength of material between exposed and unexposed samples.

## APPENDIX "D"

Resistance Measurement  
Procedure PSC 8015-4

The following procedure will be used to perform the resistance measurements required in Task I separator evaluation.

Step #1 - Preparation of Resistance Measurement

- 1) The resistivity fixture is filled with commercial grade distilled water at room ambient conditions until needed.
- 2) When the fixture is to be used, rinse with distilled water.
- 3) Rinse with 10 cc of pure MF.
- 4) Rinse with 10 cc of 3 M LiAsF<sub>6</sub>/MF.

Step #2 - Test Procedure

- 1) Charge tube with 75 cc of 3 M LiAsF<sub>6</sub>/MF.
  - a) Stoppers are placed in both elbows as in the center portion, and hoses are interconnected to equalize the overpressure in each section.
- 2) Place fixture in constant temperature environment and allow to stabilize before taking initial reading.
- 3) Measure conductivity using the conductivity bridge, Model RC 118, Industrial Instruments. Record the reading obtained and the temperature at which it was taken.
- 4) Take sample to be tested and place between fiber glass frames.
- 5) Place sample into resistivity fixture and record reading. Record temperature reading.

## Step #3

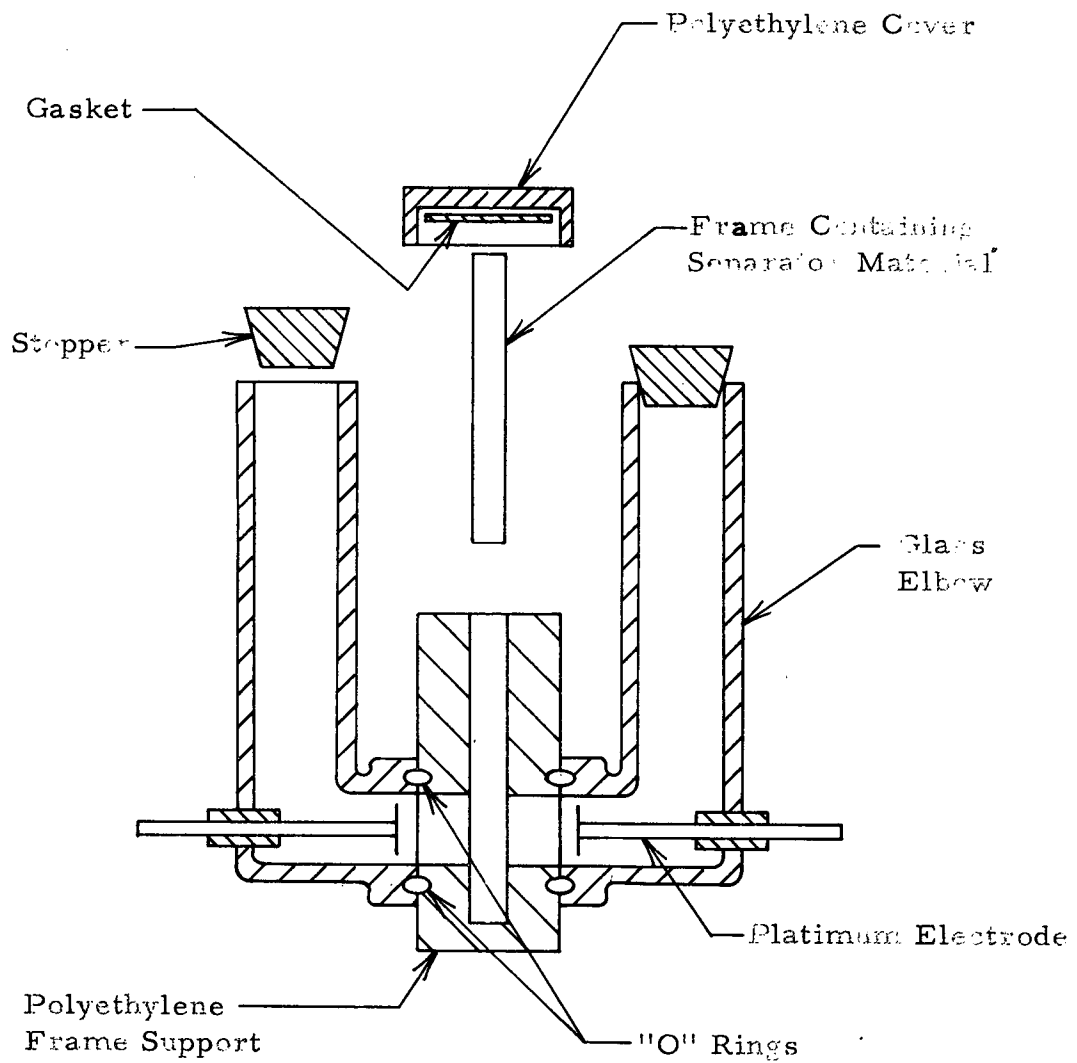
- 1) The difference between the two readings is the resistance attributed to the effect of the separator materials.

- 2) The reading on the electrolyte is taken before each sample is inserted. The volume is also checked for proper level.
- 3) This reading and the temperature is recorded for each sample tested.
- 4) At the conclusion of testing, the apparatus is cleaned with distilled water and then stored with distilled water in it.

Cell Constant:

A cell constant is established on a weekly basis by using the following procedure:

- 1) A standard mixture of 0.1 n KCl is used to establish the cell constant.
  - a) The fixture is prepared by first rinsing with 10 cc of 0.1 n KCl.
  - b) The fixture is then charged with 75 cc of the standard mixture of 0.1 n KCl.
  - c) Place into constant environment chamber and allow to stabilize for 30 minutes.
  - d) Measure the conductivity using the Model RC 118 bridge.
    - 1) Take five independent readings. (Independent defined as all instrument dials set to zero and obtaining a reading without changing the 0.1 n KCl solution.)
    - 2) Record temperature after each reading.
  - e) Average the five readings and obtain the handbook value for conductivity of 0.1 n KCl at this temperature.
    - 1) Use the standard conductivity relationship to calculate the cell constant and record.



FORM FM-100

FIGURE D-1

## APPENDIX "E"

Failure Analysis  
Procedure PSC 8015-5

The following procedure will be used to evaluate the cells undergoing tests outlined in Task II of NASA Contract No. NAS 3-13221.

- Step #1 - At the conclusion of the test, a voltage reading is taken and recorded on the Failure Analysis sheet.
- Step #2 - A sample of the gas within the chamber is obtained for analysis if desired.
- Step #3 - The test chamber is then dismantled, the electrolyte remaining checked and the amount of excess is estimated as well as any changes in color as physical appearance.
- Step #4 - The anode and cathode are checked for resistance across the connection points.
- Step #5 - The cell is removed from the test chamber by cutting screens near connection points.
- Step #6 - General appearance of the anode is noted. Specific comments concerning amount of reaction, pattern of reaction, and color of lithium/base screen.
- Step #7 - The separator is now checked for any color or physical changes and notations made.
- Step #8 - The cathode is checked for general appearance including deposits of copper, pattern of plating, liquid saturation, coloration of surface and base screen. Notations are made.
- Step #9 - Any unusual condition present in the cell is also noted.
- Step#10 - Components are then placed in the proper disposal containers. \*

\* There is on occasion additional chemical or physical tests that may be required, depending on the nature of the specific cell being analyzed. The technician must check with program engineering in this respect before performing Step 10.

Post Test and Failure Analysis

Test No: \_\_\_\_\_

Type of Test \_\_\_\_\_

Date \_\_\_\_\_

Type of Failure - If Any:

Gas Analysis \_\_\_\_\_

Comments: \_\_\_\_\_

Electrolyte

Electrolyte \_\_\_\_\_

General Appearance \_\_\_\_\_

Color \_\_\_\_\_

AnodeResistance \_\_\_\_\_  $\Omega$ 

General Appearance \_\_\_\_\_

Comments: \_\_\_\_\_

Separator

Color Change \_\_\_\_\_

Physical Change \_\_\_\_\_

General Appearance \_\_\_\_\_

Cathode

Resistance \_\_\_\_\_ ohms

General Appearance of Surface \_\_\_\_\_

Comments: \_\_\_\_\_

FIGURE E-1



## APPENDIX "F"

### Screen Cleaning Procedure PSC 8015-6

The following procedure will be used to prepare the component screens.

Step #1 - Cut screen to the appropriate size (one of the following).

1.875 x 2.875  
2.625 x 3.875  
3.375 x 5.125

Step #2 - After being cut, the screen will be cleaned by dipping into the following baths:

- 1) 10% HCl for 2 minutes
- 2) Distilled H<sub>2</sub>O for 1 minute
- 3) 10% HNO<sub>3</sub> for 2 minutes
- 4) Distilled H<sub>2</sub>O for 1 minute
- 5) Pure Acetone for 1 minute

Step #3 - Air dry at room ambient (dry room) for 5 minutes.

Step #4 - Weigh accurately to four places.

Step #5 - Place screen in polyethylene bag and record on a tag the following information:

- 1) Log number of screen.
- 2) Weight of screen.
- 3) Size and Mesh Designation.
- 4) Date cleaned.

Step #6 - Record in master log.

## Base Screen Log Sheet

[illegible]

FIGURE F-1

## APPENDIX "G"

Preparation of Polystyrene/Xylene Solutions  
 Procedure PSC 8015-7

- Step 1 - Constituent materials are obtained from the NASA storage locker.  
 They are:

Styrene pellets - packaged in labeled quart jars.  
 Xylene - MCB analyzed reagent.

Both components should be in properly numbered and labeled containers; otherwise they are not to be used. The bottle numbers are logged on the polystyrene/xylene log (F-11).

- Step 2 - The solutions are made up in approximately 500 - cc quantities. One pint of xylene/batch is sufficient.
- Step 3 - 425 ml of xylene are used; this weighs 365.9 grams (SpG. xylene = 0.861).
- Step 4 - Depending on the percent styrene solution desired, the following calculations are made to determine the weight of styrene pellets to be used. Example - 5% styrene solution desired.

$$\text{Weight styrene} = \frac{.05 (365.9)}{1 - .05} = 19.3 \text{ grams styrene pellets}$$

$$\text{General: Weight styrene} = \frac{(\text{weight fraction styrene}) (\text{weight xylene})}{1 - (\text{weight fraction styrene})}$$

- Step 5 - The 425 ml of xylene are measured and entered into a clean, dry 500 ml bottle.
- Step 6 - The amount of styrene determined in step 4 is weighed out to nearest 0.1 gram and placed in the bottle containing the 425 ml xylene.
- Step 7 - The bottle is sealed with a syringe stopper and adhesive tape and placed on a shaker overnight.
- Step 8 - The following morning the bottle is vented with a syringe needle and the solution is checked visually for complete dissolving of the pellets. If they are not dissolved, the bottle is returned to shaker.

Step 9 - The bottle is labeled as follows:

Polystyrene/Xylene  
\_\_\_\_\_ % Weight  
Date Manufactured \_\_\_\_\_  
NAS 3-13221  
Bottle No. \_\_\_\_\_

The proper bottle number is found by checking form F-11.

Step 10 - The specific gravity of the solution is determined (two samples 10 cc/sample) and the average value entered in F-11.

Step 11 - The water content is determined by Karl Fischer analysis (procedure 8015-2) and entered on F-11.

Step 12 - The grams of styrene/cc solution is determined using the specific gravity value and entered on F-11.

$$\text{Sp. Gravity} \times \text{Weight Fraction Styrene} = \frac{\text{Gram Styrene}}{\text{cc Solution}}$$

Step 13 - Necessary calculations are recorded in assigned Honeywell notebook.

Step 14 - The results of steps 10, 11 and 12 are reviewed by engineer in charge, and if acceptable the bottle is given a blue tag and stored in the processed section of the NASA storage cabinet. If the solution is not acceptable, and cannot be adjusted to meet requirements, it is disposed of at earliest convenience.



## APPENDIX "H"

Activation and Test  
Procedure PSC 8015-8Preparation:

- Step 1: Retrieve previously manufactured cell components from their storage area in the dry room and assemble according to the experimental design and place into the cell test chambers.
- Step 2: Pressurize each cell chamber with Argon (50 psi) and allow to stand overnight. Check units for any leakage occurring and apply clamps if necessary to any units indicating a drop in pressure of > 5 psi.
- Step 3: Turn on environmental chambers and allow to stabilize at the designated temperature checking all thermocouples to verify that the monitoring channels are functioning on the recorder.
- Step 4: (a) Transport cells to be tested to the environmental lab and by referring to the test sheets, make up tags of the required electrolyte.
- (b) Note on the tag the cell number, test date, electrolyte batch number, molarity of electrolyte, and the amount of electrolyte required.
- Step 5: From the electrolyte area, obtain the designated volume and molarity of electrolyte, in lecture bottles for each cell. (If SO<sub>2</sub> is to be used the proper ratio of SO<sub>2</sub>/gram of electrolyte is added to the lecture bottle.
- Step 6: Release the Argon pressure from the cell test chamber and attach the safety release valve which has been previously checked at 80 psig.
- Step 7: (a) Place cell test chamber into the environmental chamber onto styrofoam pads, which keeps condensed moisture from close contact with the cell chambers and keeps clamped cells level.
- (b) Insert thermocouple probe into cell test chamber, \* making certain thermocouple for the environmental chamber is not

\* Not used with glass tubes.

touching the battery or side and is protected from direct draft from blower.

(c) Label outside of environmental chamber with the cell number.

(d) Cover relief valve with plastic bag and clamp on outside of environmental chamber.

Step 8: Record date, voltage recorder channel numbers, and thermocouple numbers on test sheet, (Form F-17).

Step 9: Place filled electrolyte lecture bottles into appropriate chambers and allow a minimum of one-half hour for conditioning of both cell and electrolyte to temperature before activating.

Step 10: Begin calibration.

#### Calibration:

Step 1: (a) Turn on transducer and allow five (5) minutes warm up time. Meanwhile connect argon tank and pressure calibration guage and check chamber temperatures.

(b) Use a chart speed of 50 inches/hour during calibration. By adjusting the argon pressure, check the transducer over a range of 0 to 80 lbs per square inch. Adjust zero reading and recalibrate if needed. Return dial to 4 inches/hour when finished and stop chart. Mark chart with date, 0, 20, 40, 60, and 80 psi readings and speed.

Turn off argon.

(c) Connect argon line to pressure guage used during activation.

Step 2: (a) Attach standard dry cell battery to voltage recorder.

(b) Turn on recorder and attach calibrated voltmeter (Digitest) to check readings. Mark date, voltage reading, and speed (10 inches/hour) on chart. Read voltage for 5 minutes on one odd and one even set of channels. Put recorder on stand-by until activation of cells.

- Step 3: Connect resistance lead to the positive terminal and both positive and negative leads of voltage recorder to cell under test when calibration is completed. Place negative lead from resistor inside environmental chamber but do not connect. Make sure it does not touch negative terminal.

Activation - Standard NASA Cell:

- Step 1: Connect volt-meter (Digitest) to first cell to be tested and turn "on".
- Step 2: (a) Connect vacuum line to the cell at the valve at pressure guage and pull a vacuum of 500 microns or lower.
- (b) Insert needle of hose from lecture bottle through the diaphragm of the activation port in cell chamber and bring vacuum to 500 microns again. Plastic hose may collapse.
- Step 3: (a) Adjust argon pressure so as to permit applying a nominal 10 pounds of pressure to top of lecture bottle.
- (b) Connect argon line to top valve of lecture bottle, keep valve on bottle closed.
- Step 4: Start voltage recorder.
- Step 5: Close valve on cell, and turn "off" vacuum line and disconnect.
- Step 6: (a) Open top valve, then bottom valve on electrolyte lecture bottle allowing the liquid to flow into the cell. Start clock and record activation time on test data sheet when voltmeter registers voltage.
- (b) Allow all the electrolyte to flow into cell and let 10 pounds of argon pressure to accumulate in cell.
- (c) Remove needle from cell and close both valves on the lecture bottle.
- (d) Replace pressure cap on activation port of cell chamber and place cap on valve at pressure guage.
- (e) Turn "off" argon and disconnect electrolyte lecture bottle.



NOTE: The following steps apply unless unit is placed on active stand where OCV is recorded daily until end of stand period, then steps 9 to 18 are followed.

- Step 7: If more than one cell is to be activated repeat steps 1 through 7 at 4 minute intervals. Total of 3 cells can be activated in a single sequence.
- Step 8: Mark cell number and monitoring channels on voltage chart.
- Step 9: (a) Record open circuit voltage (OCV) ten minutes after activation.
- (b) Connect variable resistor and run polarization scan, switching the resistance from 100 ohms to 1 ohm through steps of 100, 50, 25, 10, 5, 4, 3, 2, and 1 ohm. Record voltage as each resistance is applied and after 15 seconds.
- Step 10: If a transducer is to be applied, connect it after the polarization scan. Turn "on" chart, but do not open valve until load is connected to negative terminal.
- Step 11: (a) At 20 minutes after activation, record OCV and attach resistor to the negative terminal of the cell. Open valve to transducer and check ammeter reading to verify a closed circuit.
- (b) Record initial closed circuit (ICV) and time of load on test data sheet.
- (c) Mark on the temperature chart recorder the cell number and channel.
- (d) Mark transducer chart with cell number.
- Step 12: Return the lecture bottle to the electrolyte area for cleaning.
- Step 13: (a) Check periodically on temperature of environmental chamber to be sure it remains constant.
- (b) Check cell voltage, any sudden drop in voltage and an increase in cell temperature (more than 10° or 12°F above environment) probably indicates a short circuit condition and cell should be disconnected.

- Step 14: (a) Allow cell to run until voltage reaches 1.9 volts or for low potential cells 0.9 volts.
- (b) Record final pressure, and maximum closed circuit voltage (MCV) obtained.
- Step 15: (a) To disconnect cell after discharge turn "off" environmental chamber.
- (b) Disconnect voltage recorder, thermocouple, and resistor.
- (c) If NO gas sample is to be taken, remove cap on valve at pressure guage and bleed the unit. Release pressure to zero reading on guage, then close valve and replace cap.
- Step 16: If gas sample is to be taken, prepare sample bottle prior to end of discharge by pulling a vacuum of 200 microns. Connect sample bottle to cell with shortest possible tube. Vacuum the inter connecting tube. When it is  $\leq 200 \mu$ , close vacuum and open valve to cell chamber allowing cell gas to enter sample tube.
- (b) Record pressure of cell before and after sample is taken and pressure indicated on sample bottle if any.
- (c) Label gas sample and cap it.
- (d) Bleed remaining pressure from cell.
- Step 17: Fill out postmortem sheet and place with cell in the postmortem area. Leave safety valve connected and notify the P.M. technician that the cell is ready.
- Step 18: Turn "off" transducer and voltage recorder. Remove charts for cells discharged and remove temperature chart.

#### Activation - Glass Tube:

- Step 1: (a) Connect vacuum line to the cell at pressure gauge and pull a vacuum of 500 microns or lower.
- (b) Close valve and remove vacuum line. Place serum stopper over valve port.
- Step 2: (a) With syringe, remove proper amount of electrolyte from bottle.

(b) Insert needle of syringe into serum stopper and open valve until all the electrolyte flows into cell.

(c) Remove needle from cell, close valve and remove serum stopper from port.

(d) If  $\text{SO}_2$  is to be added, the correct ratio per gram of electrolyte is added at this point.

Step 3: (a) Record open circuit voltage (OCV) 10 minutes after activation -- Steps 9 to 18 of the standard cell procedure, with the exception of Step 12, apply as stated either immediately or after designated stand period.

## APPENDIX "I"

Cathode Fabrication  
Procedure PSC 8015-9

The following covers the general procedure and material control considerations used in the manufacturing of pasted cathodes.

1. A listing of specific cathode requirements is generated by the engineer using the basic experiment design. These are listed on form F-12, which is then given to the technician in charge along with schedule of expected usage.
2. Using this sheet (F-12) the technician sets up the cathode assembly worksheet (F-13) which the assembler uses as a guide to manufacturing the cathode as well as making notations during the process.
  - A.  $\text{CuF}_2$   $\longrightarrow$  weight and bottle number
  - B.  $\text{CuF}_2 \cdot 2\text{H}_2\text{O}$  weight and bottle number
  - C. Carbon  $\longrightarrow$  type, weight, and bottle number
  - D. Styrene  $\longrightarrow$  % sol., vol., and bottle number
  - E. Screen number
  - F. Cathode size and thickness
    - 1) Also to be filled in by cathode fabricator--
      - a. CC of styrene solution added
      - b. Name
      - c. Date
      - d. Drying time in and out (at room environment)
      - e. Drying time in vacuum
      - f. Post drying weight
3. The fabricator gathers together all salts and carbon from storage and places them, with the cups and beakers and utensils required, into glove box after checking glove box for maximum of 5% oxygen content.

4. The salts and carbon are then weighed individually, placed together, and micronized for two 20-second periods. The mix is then placed in a plastic beaker and the required cc's of styrene solution are added. If the mix does not have a heavy paste consistency, add xylene drop-by-drop from a syringe and note vol. added.
5. When the proper consistency is obtained, place approximately 1/2 of it into the cathode mold which has been previously set at the proper thickness. After the mix has been spread evenly, gently impress the collector screen into it.
6. Apply the balance of the mix over the screen and level it with a spatula. Smooth the cathode a final time with a glass rod.
7. Remove the cathode from the mold and place it onto a glass plate covered with tedlar. Place another glass plate on top of the cathode. The tedlar used during fabrication is left on top of the cathode. Allow the cathode to dry in the low humidity room until end of day when all cathodes made that day are placed in vacuum for overnight drying. (Note time on worksheet.)
8. After overnight vacuum the cathodes are weighed, placed in a polyethylene bag and stored in a desiccator cabinet.
9. The completed cathode worksheet is returned to engineering for capacity calculations and final logging.

## APPENDIX "J"

Anode Fabrication  
Procedure PSC 8015-10

The following procedure and material control considerations are used in the manufacturing of anodes.

1. From the experiment sheet the percentage over 100% of the cathode capacity is determined and the anode capacity is derived. From this required capacity the configuration of the anode is compiled.
2. Anode surface area is constant; therefore, the thickness of the anode is the dimension varied to control the capacity.
3. The dimensions of the required lithium pieces to make up the needed weight are calculated by engineering and along with other pertinent data are placed on the anode worksheet.
4. On the anode worksheet are the following data:
  - A. Anode No.
  - B. Screen No.
  - C. Size
  - D. Capacity
  - E. Sheet lithium required (size in length and width)
  - F. Mold size (height)
  - G. Shims required (for compression)
  - H. Lithium weight (for capacity)
  - I. Anode thickness
5. From the lithium stock in stockroom, a sufficient quantity is withdrawn to fabricate the anodes to be made. After the as received container is opened, the lithium is stored from that point on in a desiccator containing Drierite.
6. The operator cuts lithium per worksheet instructions. The weight of these strips is then determined and compared with the desired weight on the worksheet. If in variance with this value, the strips are trimmed or additional strips added until the actual weight and desired weights are in tolerance.

7. The mold is selected according to the worksheet (height and size) along with the shims necessary for pressing the anode. .
8. The mold is opened and half the shims are placed inside. The screen is placed in the mold followed by the lithium strips. Remaining shims are placed on top of the lithium. The ram of the mold is inserted, after which the mold is placed in the press and pressed to 1000 psi. The pressure is maintained for one minute. The mold is then disassembled and the anode removed. It is then weighed, measured for thickness, packaged in a polyethylene bag, and stored in a desiccator.
9. Data generated during fabricating along with worksheets are returned to engineering for calculation of capacity and recording in log books.

## APPENDIX "K"

Cell Construction  
Procedure PSC 8015-11

The following procedure is used for the construction of cells in the standard NASA chamber or glass tubes.

1. All components of each cell are logged after manufacture in their respective log books.
2. From these books the parts are checked out for cell assembly as per test sheet of each experiment.
  - A. Each component is checked twice; once by the technician in charge, and once by the cell assembler.
3. The parts are then taken from storage and arranged in specific cell groups for build.
4. The chambers for each cell are taken off of pressure leak check, dismantled, and laid out for component insertion. From the data in the log books along with other design information, the Cell Construction Log Sheet is filled out by the technician in charge and checked by the engineer in charge. This book contains all data relative to the fabrication and testing of the cells.
- 5A. Standard NASA Chamber -

Component insertion into the standard NASA chamber is in the order of anode, separator, cathode, separator, and anode.

- 1) Place anode into chamber, cut out hole in screen for connection to terminal post. Mark the side of chamber negative corresponding to the terminal to which the anode is connected.
- 2) Lay in large separator, fold up around sides of chamber, and cut out flap for screen connection.
- 3) Lay in cathode and connect to opposite terminal post by cutting hole in screen.
- 4) Lay separator on top of cathode and fold over large separator to form envelope.
- 5) Lay in second anode and make connection to same negative post. Secure connections with plastic washer and nuts.



- 6) Take height of cell from cell construction log and subtract from depth of chamber to determine shim height.
- 7) Place shim into chamber and assemble chamber making sure the "O"-ring is lightly greased and in its groove.
- 8) The chamber is then tightened securely and put on leak check overnight. Argon pressure of 40 to 60 psi is used.

#### 5B. Cell Construction Glass Compatibility Tube -

The individual components of the cell are assembled prior to insertion into the glass tube.

- 1) Place anode on flat surface--screen side down
- 2) Place separator(s) on top of anode
- 3) Place cathode in position
- 4) Place next layer of separator(s) over cathode
- 5) Add second anode over separators.
- 6) The cell is held between two glass plates, 11/16 x 2". Secure the cell between the glass slides with two "O"-rings, equally spaced along the 2" length. The "O"-rings are Parker #2-013 compound E515-8.
- 7) Solder the two anode leads to the appropriate wires. Slide plastic insulators over the solder connections.
- 8) Solder the cathode lead to the appropriate wire. Slide a plastic insulator over the solder connection.
- 9) Insert the assembled cell into the glass tube making sure the positive and negative leads are not in danger of shorting.
- 10) Tighten gauge assembly for proper seal.
- 11) The completed cell is then charged with argon pressure of 40 to 60 psi overnight to check for leaks after which it is available for discharging.

## APPENDIX "L"

### Chemical Analyses of Materials

The following analysis was performed at the NASA - Lewis Research Center on two samples of  $\text{KAsF}_6$ . One sample was material "as received" from the vendor and the other was recrystallized according to the procedure included in Appendix "A".

TABLE L-1

$\text{KAsF}_6$  Analysis--NASA - Lewis Research Center

Description of material to be analyzed and analysis desired:

Two Samples  $\text{KAsF}_6$  Analysis for Trace Metals

Analysis Report:

Atomic Adsorption analysis  
Jarrell -Asb Spectrophotometer

Metal	$\text{KAsF}_6$ DTM-1-84	$\text{KAsF}_6$ DTM-1-84
	As Received	Batch 30 Recrystallized
	Concentration, ppm	Concentration, ppm
Al	N. D.	N. D.
Cr	0.02	0.01
Cu	N. D.	N. D.
Fe	0.3	0.18
Mg	N. D.	N. D.
Ni	5	N. D.
Ag	0.6	0.6

N. D. = Not Detected

### Mass Spectrographic Analysis

Samples of  $\text{CuF}_2$ ,  $\text{CuF}_2 \cdot 2\text{H}_2\text{O}$ ,  $\text{KAsF}_6$ , and  $\text{LiBF}_4$  were submitted "as received" to Bell & Howell Laboratories, Electronics Materials Division, Pasadena, California; and Battelle Memorial Institute, Columbus Laboratories, Columbus, Ohio, for mass spectrographic analysis.

The following charts list the results of these analyses:

TABLE L-2

Impurity Concentrations in Honeywell Cupric Fluoride As Reported by Bell & Howell  
(In Parts Per Million By Weight)

Element <sup>1</sup>	Detection Limit <sup>2</sup>	CuF <sub>2</sub>	CuF <sub>2</sub> ·2H <sub>2</sub> O <sup>3</sup>
H	0.1	2.3	1
Li	0.01	0.021	0.29
B	0.1	0.3	10
C	0.1	520	1,100
N	0.1	40	79
O	0.1	14,000	1
Na	0.03	1,400	62
Mg	0.3	8.7	27
Al	0.3	6.4	19
Si	20	57	N. D.
P	0.7	1	100
S	0.7 <sup>4</sup>	56	920
Cl	0.7	33	330
K	0.1	68	20
Ca	0.7	9.7	66
V	1	2	20
Cr	1	360	160
Mn	1	23	33
Fe	2	350	250
Co	2	35	150
Ni	3	49	61
Zn	3	45	410
Ga	2	11	14
As	3	150	580
Y	2	4.8	17
Zr	2	2.3	60
Sb	3	N. D.	160
Ba	2	N. D.	2.4
Ce	3	N. D.	15
Pb	5	N. D.	61

TABLE L-2 (Continued)

<sup>1</sup>Analyses for thorium and uranium were not made. Analyses for tantalum and gold are not given since tantalum slits are used in the mass spectrometer and the samples were sparked against high purity gold probes. The analysis of  $\text{CuF}_2 \cdot 2\text{H}_2\text{O}$  does not include hydrogen or oxygen since these are matrix elements. Other impurities not listed were not detected and have concentrations less than 5 ppmw.

<sup>2</sup>Determined for  $1 \times 10^{-7}$  coulomb exposure.

<sup>3</sup>Detection limits are three times those given.

<sup>4</sup>Detection limit for sulfur in  $\text{CuF}_2 \cdot 2\text{H}_2\text{O}$  is 30 times that listed due to interference from  $\text{O}_2$ .

N. D. = Not Detected

TABLE L-3

Impurity Concentrations in Honeywell Lithium Fluoroborate As Reported by Bell & Howell  
(In Parts Per Million By Weight)

Element <sup>1</sup>	Detection Limit <sup>2</sup>	LiBF <sub>4</sub>
H	0.1	9.6
C	0.1	9200
N	0.1	41
O	0.1	5700
Na	0.02	220
Mg	0.1	65
Al	0.5	400
Si	7	30
S	10	20
Cl	1	66
K	0.05	48
Ca	1	64
Cr	2	5.3
Fe	2	5.4
Co	2	45
Ni	2	3.5
Cu <sup>3</sup>	2	15
Zn	3	3300
As	2	410
Se	3	11
Br	3	5.1
Te	5	130
Ba	3	66

<sup>1</sup> Analyses for thorium and uranium were not made. Analyses for tantalum and gold are not given since tantalum slits were used in the mass spectrometer and the sample was sparked against a high purity gold probe. Other impurities not listed were not detected and have concentrations less than 5 ppmw.

<sup>2</sup> Determined for  $1 \times 10^{-7}$  coulomb exposure.

<sup>3</sup> May be due to residuals in the mass spectrometer.

TABLE L-4

Impurity Concentrations in Honeywell Potassium Fluoroarsenate  
As Reported by Bell & Howell (In Parts Per Million By Weight)

Element <sup>1</sup>	Detection Limit <sup>2</sup>	KAsF <sub>6</sub>
H	0.05	3.0
Li <sup>3</sup>	0.02	4.0
B <sup>3</sup>	0.1	230
C	0.1	650
N	0.1	26
O	0.1	360
Na	0.02	47
Mg	1	N. D.
Al	0.1	2.4
Si	0.2	64
S	0.2	59
Cl	0.3	49
Ca	2	N. D.
Cr	0.7	14
Fe	1	11
Ni	3	29
Cu <sup>3</sup>	2	51
Zn	3	930
Ga	2	17
Se	3	22
Sb	5	24

<sup>1</sup> Analyses for thorium and uranium were not made. Analyses for tantalum and gold are not given since tantalum slits are used in the mass spectrometer and the sample was sparked against a high purity gold probe. Other impurities not listed were not detected and have concentrations less than 7 ppma.

<sup>2</sup> Determined for  $1 \times 10^{-7}$  coulomb exposure.

<sup>3</sup> May be due to residuals in the mass spectrometer.

N. D. = Not Detected



TABLE L-5

Mass Spectrographic Analysis of Copper Fluoride (ppmw)  
As Reported by Battelle Memorial Institute

Element	Sample Number		Element	Sample Number	
	#7	#9		#7	#9
Li	0.1	0.1	Cd	<1.	<1.
Be	<0.01	<0.01	In	<0.06	<0.6
B	0.06	0.02	Sn	<1.	<1.
Na	30.	300.	Sb	2.	<0.4
Mg	40.	10.	Te <sup>2</sup>	<15.	<15.
Al	4.	4.	I	5.	<0.5
Si	5.	5.	Cs	<0.6	<0.1
P <sup>1</sup>	<10.	<10.	Ba	2.	<0.2
S	20.	10.	La	<0.1	<0.5
Cl	20.	20.	Ce	50.	<1.
K	3.	1.	Pr	3.	<1.
Ca	300.	6.	Nd	<2.	<2.
Sc	<1.	<1.	Sm	<0.3	<0.3
Ti	2.	<0.2	Eu	<0.2	<0.2
V	0.4	<0.04	Gd	<0.3	<0.3
Cr	10.	3.	Tb	<0.1	<0.1
Mn	10.	10.	Dy	<0.3	<0.3
Fe	300.	60.	Ho	<0.3	<0.3
Co	<1.	<3.	Er	<0.3	<0.3
Ni	10.	20.	Tm	<0.1	<0.1
Zn	20.	6.	Yb	<0.3	<0.3
Ga	<1.	<1.	Lu	<0.1	<0.1
Ge	<0.4	<0.4	Hf	<0.3	<0.3
As	<4.	<1.	Ta <sup>3</sup>	<3.	<3.
Se	<2.	<2.	W	<0.3	<0.3
Br	<1.	<3.	Re	<0.2	<2.
Rb	<1.	<1.	Os	<0.3	<0.3
Sr	10.	<0.5	Ir	<0.2	<0.6
Y	15.	<0.5	Pt	<0.3	<1.
Zr	<0.3	<0.6	Au	<1.	<1.
Nb	<0.04	<0.4	Hg	<0.4	<0.4
Mo	<0.6	<0.6	Tl	<0.5	<0.5
Ru	<0.2	<0.5	Pb	10.	10.
Rh	<0.4	<1.	Bi	<0.4	<0.4
Pd	<0.6	<2.	Th	<0.4	<0.4
Ag	<7.	<7.	U	<0.4	<0.4

<sup>1</sup> Interference from CF<sup>+</sup>

<sup>2</sup> Interference from Cu<sub>2</sub><sup>+</sup>

<sup>3</sup> Uncertainty due to possible contribution from Ta electrode holders

TABLE L-6

Mass Spectrographic Analysis of  $\text{KAsF}_6$  and  $\text{LiBF}_4$  (ppmw)  
As Reported by Battelle Memorial Institute

Element	Sample Designation		Element	Sample Designation	
	$\text{KAsF}_6$	$\text{LiBF}_4$		$\text{KAsF}_6$	$\text{LiBF}_4$
Li	$\leq 10$ <sup>1</sup>	-	Cd	$< 5$	5
Be	$< 0.005$	0.1	In	$< 0.2$	$< 0.6$
B	$\leq 15$ <sup>1</sup>	-	Sn	$< 2$	$\leq 2$
Na	3	30	Sb	3	$< 1$
Mg	$< 1$	15	Te	$< 0.6$	$< 1$
Al	$\leq 1$	$\leq 2$	I	$< 0.2$	$< 0.6$
Si	10	$< 10$	Cs	$< 1$	$< 1$
P	$\leq 15$	$\leq 3$	Ba	$< 0.3$	$< 1$
S	2	6	La	$< 0.1$	$< 1$
Cl	10	10	Ce	$< 0.1$	$< 0.6$
K	-	10	Pr	$< 0.2$	$< 0.6$
Ca	10	60	Nd	$< 3$	$< 3$
Sc	$< 0.2$	$< 2$	Sm	$< 1$	$< 3$
Ti	$< 2$	$< 10$	Eu	$< 0.3$	$< 2$
V	$< 0.3$	$< 3$	Gd	$< 0.3$	$< 1$
Cr	$\leq 0.1$	$< 0.3$	Tb	$< 0.3$	$< 0.3$
Mn	$< 0.3$	0.3	Dy	$< 0.4$	$< 1$
Fe	1	5	Ho	$< 0.3$	$< 1$
Co	$< 0.3$	$< 3$	Er	$< 0.6$	$< 2$
Ni	$< 0.05$	0.5	Tm	$< 0.3$	$< 0.3$
Cu	0.6	0.3	Yb	$< 1$	$< 1$
Zn	$< 0.02$	$\leq 2$	Lu	$< 0.3$	$< 0.3$
Ga	$< 0.3$	$< 1$	Hf	$< 0.4$	$< 1$
Ge	$< 0.4$	$< 1$	Ta	$\leq 2$	$< 1$
As	-	$\leq 4$	W	$< 0.3$	$< 1$
Se	$\leq 3$	$< 1$	Re	$< 0.1$	$< 1$
Br	$< 1$	$< 1$	Os	$< 0.3$	$< 1$
Rb	$< 20$	$< 0.4$	Ir	$< 0.3$	$< 0.6$
Sr	$\leq 2$	$\leq 0.4$	Pt	$< 0.2$	$< 1$
Y	$< 1$	$< 1$	Au	$< 0.1$	$< 0.3$
Zr	$< 0.3$	$< 0.3$	Hg	$< 0.1$	$< 1$
Nb	$< 5$	$< 0.5$	Tl	$< 0.4$	$< 0.4$
Mo	$< 5$	$< 2$	Pb	$< 0.2$	$< 1$
Ru	$< 0.5$	$< 0.5$	Bi	$< 0.05$	$< 0.1$
Rh	$< 0.5$	$< 0.2$	Th	$< 0.1$	$< 0.3$
Pd	$< 2$	$< 0.6$	U	$< 0.1$	$< 0.3$
Ag	$< 1$	$< 3$			

<sup>1</sup> Possible memory from previous sample ( $\text{LiBF}_4$  run first).

## APPENDIX "M"

List of Materials and Suppliers

Material	Description	Use	Supplier
1. Copper Fluoride ( $\text{CuF}_2$ )	Anhydrous double HF treated	Cathode material	Ozark-Mahoning Co. 310 W. Sixth Avenue Tulsa, Okla.
2. Copper Fluoride Di- hydrate ( $\text{CuF}_2 \cdot 2\text{H}_2\text{O}$ )		Cathode	Ozark-Mahoning Co. 310 W. Sixth Avenue Tulsa, Okla.
3. Graphite	Asbury Ceylon Graph- ite #280	Cathode conductor ma- terial	Asbury Graphite Mills, Inc. Asbury, N.J.
4. Graphite	Dixon Air Spun Graph- ite (94-95%) Carbon Content	Cathode conductor ma- terial	Joseph Dixon Crucible Co. Jersey City, N.J. 07303
5. Graphite	Conductex SC uncom- pressed	Cathode conductor ma- terial	Columbian Carbon Co. 380 Madison Avenue New York, N.Y. 10017
6. Styrene	Styron Polystyrene	Cathode binder mater- ial	Dow Chemical Corporation Midland, Mich.
7. Xylene	XX55-Reagent Gra	Cathode binder solvent	Matheson, Coleman & Bell East Rutherford, N.J.
8. Potassium Hexafluoro- arsenate ( $\text{KAsF}_6$ )		Salt used in electrolyte preparation	Ozark-Mahoning Co. 310 W. Sixth Avenue Tulsa, Okla.
9. Lithium Tetrafluoro- borate ( $\text{LiBF}_4$ )		Salt used in electrolyte preparation	Foote Mineral Route 100 Exton, Penna. 19341

Material	Description	Use	Supplier
10. Lithium Hexafluoroarsenate ( $\text{LiAsF}_6$ )		Salt used in electrolyte preparation	U. S. Steel Agri-Chemical Division 685 DeKalb Industrial Way Decatur, Ga. 30030
11. Methyl Formate ( $\text{HCOOCH}_3$ )	Spectroquality grade	Solvent used in electrolyte preparation	Matheson, Coleman & Bell East Rutherford, N.J.
12. Li Metal Powder	Battery grade 10 micron average particle	Electrolyte preparation	Foote Mineral Co. Route 100 Exton, Penna. 19341
13. Li Metal	Ribbon argon packed	Anode material	Foote Mineral Co. Route 100 Exton, Penna. 19341
14. Exmet (Distex) Collector	Expanded metal screen	Anode & cathode collector grids	Exmet Corporation 355 Hanover Street Bridgeport, Conn. 06601
15. Glass Fiber Filter	Grade 934H	Candidate separator material	H. Reeve Angel & Co., Inc. 9 Bridewell Place Clifton, N.J. 07014
16. Pellon	FT-2140 nonwoven polypropylene	Candidate separator material	Pellon Corporation 491 Dulton Street Lowell, Mass. 01852
17. Porvic	Microporous polyvinyl chloride	Candidate separator material	Porvair Ltd. Estuary Road King's Lynn-Norfolk, England

Material	Description	Use	Supplier
18. Weblox EV10A	100% Dacron	Candidate separator material	Kendall Fiber Products 12 South Twelfth Street Philadelphia, Penna. 19107
19. Molecular Sieve	Grade 4A	Purification of MF solvent	Union Carbide Corporation Linde Division Pleasant Valley Road/ Highway 38 Moorestown, N. J. 08057